



**US Army Corps  
of Engineers**  
South Atlantic Division



Region 4

**Regional Implementation Manual**

**Requirements and Procedures  
for Evaluation of the Ocean Disposal of Dredged Material  
in Southeastern Atlantic and Gulf Coastal Waters**

May 1993

The U.S. Environmental Protection Agency and the U.S. Army Corps of Engineers published national guidance on procedures to be followed when assessing the suitability of dredged material for disposal in the ocean. This guidance is entitled: Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual (56 Federal Register 13826, April 4, 1991) and requires the development of Regional Implementation Agreements for activities regulated under Section 103 of the Marine Protection, Research and Sanctuaries Act of 1972 (33 USC 1401 et seq.).

This Regional Implementation Manual complies with the EPA/CE national guidance specified in the 1991 Green Book and has been approved by the following officials of EPA and CE, and goes into effect upon the date of the last signature:

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Greer Tidwell  
Regional Administrator  
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## **List of Abbreviations and Acronyms**

<b>Abbreviations and Acronyms</b>	<b>Definition</b>
ADDAMS	Automated Dredging and Disposal Alternatives Management System
ANOVA	Analysis of variance
CE	U.S. Army Corps of Engineers
CFR	Code of Federal Regulations
COD	Chemical Oxygen Demand
CRM	Certified Reference Material
DO	Dissolved oxygen
EMAP	Environmental Monitoring and Assessment Program
EPA	U.S. Environmental Protection Agency
FDA	U.S. Food and Drug Administration
GC/MS	Gas chromatograph/mass spectrophotometer
HMW	High molecular weight
l	liter
LCM	Laboratory Control Material
LOEL	Lowest Observed Effect Level
LPC	Limiting Permissible Concentration
MDL	Method detection limit
mg/kg	milligrams/kilogram or parts per million
ml	milliliter
MOU	Memorandum of Understanding
mm	millimeters
MPRSA	Marine Protection Resources Sanctuaries Act

ng/kg	nanograms/kilogram or parts per trillion
NIST	National Institute of Standards and Technology
NOAA	U.S. National Oceanographic and Atmospheric Administration
NRCC	National Research Council of Canada
NS&T	National Status and Trends Program
ODMDS	Ocean Dredged Material Disposal Site
PAH	Polynuclear Aromatic Hydrocarbons
PCB	Polychlorinated Biphenyls
PCT	Polychlorinated Terphenyls
ppt	parts per thousand
QA	Quality Assurance
QC	Quality Control
RIM	Regional Implementation Manual
RPD	Relative Percent Difference
SAD	South Atlantic Division
TBP	Theoretical Bioaccumulation Potential
TIC	Total Inorganic Carbon
TKN	Total Kjeldahl Nitrogen
TOC	Total Organic Carbon
$\mu\text{E}/\text{m}^2/\text{sec}$	micro Einsteins per meter square per second
Tg/l	micro grams per liter or parts per million
WQC	Water Quality Criteria

## 1.0 INTRODUCTION

The potential adverse effects from the ocean disposal of dredged material in the marine environment can range from unmeasurable to significant. These effects may vary depending on many factors, including the composition of the proposed dredged material (e.g., the presence of contaminants, sediment grain size, etc.) and disposal site location. As a result, dredging and disposal operations are evaluated on a case by case basis. Federal regulations require such evaluations, with emphasis on potential biological impacts from the disposal of dredged material in the marine environment. According to Section 103 of the Marine Protection, Research, and Sanctuaries Act of 1972 (MPRSA), any proposed placement of dredged material in the ocean waters of the United States must be evaluated according to the criteria published by the U.S. Environmental Protection Agency (EPA) in Title 40 of the Code of Federal Regulations (CFR), Parts 220-228. The actual evaluation is conducted by the U.S. Army Corps of Engineers (CE) which is the permitting agency for the transportation of dredged material to the ocean for the purpose of disposal, subject to EPA review and concurrence. MPRSA and Part 225 allow a waiver of the criteria, in extreme cases, if the proposed action is denied by EPA, but dredging is essential and feasible alternatives are unavailable. Only the EPA Administrator may grant such waivers [40 CFR Part 225.4].

National guidance for the evaluation of dredged material under MPRSA Section 103 program is provided in the "Evaluation of Dredged Material Proposed for Ocean Disposal - Testing Manual" (EPA and CE, 1991). This manual, more commonly known as the "1991 Green Book", includes a description of the tiered approach to sediment testing. Included in the manual are methods and procedures for sediment sampling and testing, general guidance on bioassay and bioaccumulation testing, as well as an overview of data analyses and quality control/assurance procedures. The 1991 Green Book supersedes the 1977 Green Book (EPA and CE, 1977).

In July 1990, the CE South Atlantic Division and the EPA Region IV signed a Memorandum of Understanding (MOU). This MOU is intended to clarify CE and EPA responsibilities with respect to the implementation of MPRSA, as amended. This Regional Implementation Manual (RIM) is an outgrowth of that MOU, and represents an agreement between the EPA and the CE Districts within Region IV for the use of the 1991 Green Book. This RIM documents testing and reporting requirements for the ocean disposal of dredged materials along the southeastern and gulf coasts of the United States. This agreement is based on regulatory requirements under Section 103 of the MPRSA of 1972. These requirements apply to all permit applicants and civil works projects which are subject to the criteria defined in EPA's Ocean Dumping Regulations in 40 CFR Parts 225 and 227.

Additional information may be required, depending on the nature and location of the proposed project. In most cases, the project will also need to satisfy state regulatory requirements.

CE Districts will provide a complete package, compiled from all available information, to EPA Region IV and other pertinent regulatory agencies for review and comment. This information will serve as the basis for a determination of permit issuance and/or subsequent enforcement, if necessary, under MPRSA Sections 105 and 107.



This RIM provides the EPA Region IV, CE, state regulatory agencies, permit applicants, and other interested parties with detailed information on federal regulatory requirements and coordination procedures for the ocean disposal of dredged material within the CE South Atlantic Division (SAD) and EPA Region IV. Information in this RIM includes the following:

- A. Program Coordination
- B. Administrative Requirements
- C. Tiered Testing and the 1991 Green Book
- D. Sediment Sampling
- E. Physical and Chemical Testing
- F. Bioassay and Bioaccumulation Testing
- G. Statistical Analyses
- H. Sediment Testing Report Format
- I. Quality Control and Quality Assurance

New information is continually being developed by the Ocean Dredged Material Disposal Program. This information includes: new regulations, national program guidance, dredging and disposal management operations, as well as scientific improvement in sediment testing procedures. When these new developments warrant changes in procedures, this RIM will be updated. Clarifications and questions pertaining to this manual should be directed to EPA Region IV or the appropriate CE District offices (Appendix A).

Copies of the EPA/CE 1991 Green Book are available by writing to the:

U.S. Army Corps of Engineers  
Waterways Experiment Station, EP-D  
3909 Halls Ferry Road  
Vicksburg, Mississippi 39180  
(601)-634-3701

## **2.0 EPA/CE OCEAN DISPOSAL PROGRAM COORDINATION**

### **2.1 General Principles**

The CE Districts and EPA Region IV work cooperatively in the management of the Ocean Dredged Material Disposal Program to ensure that each agencies responsibilities are met. Coordination occurs through formal review processes (Figure 2.1) and informal staff communications. Should concern arise, the EPA and CE District will resolve identified problems as early as possible to avoid potential project delays. Consequently, information critical to determinations regarding the suitability of dredged material for ocean disposal is required by the CE District and EPA Region IV at the earliest reasonable time. Appendices B and C describe this information. All coordination with EPA Region IV for activities involving ocean disposal of dredged material is the responsibility of the respective CE District Office.

There are two points in the MPRSA 103 evaluation process where coordination and communication are important to project success: (1) the need for testing determinations (i.e., exclusionary criteria and test plan development) ; and (2) the MPRSA Section 103 evaluation determination. The following sections describe the needed information and time lines for EPA and the CE District for these two coordination points.

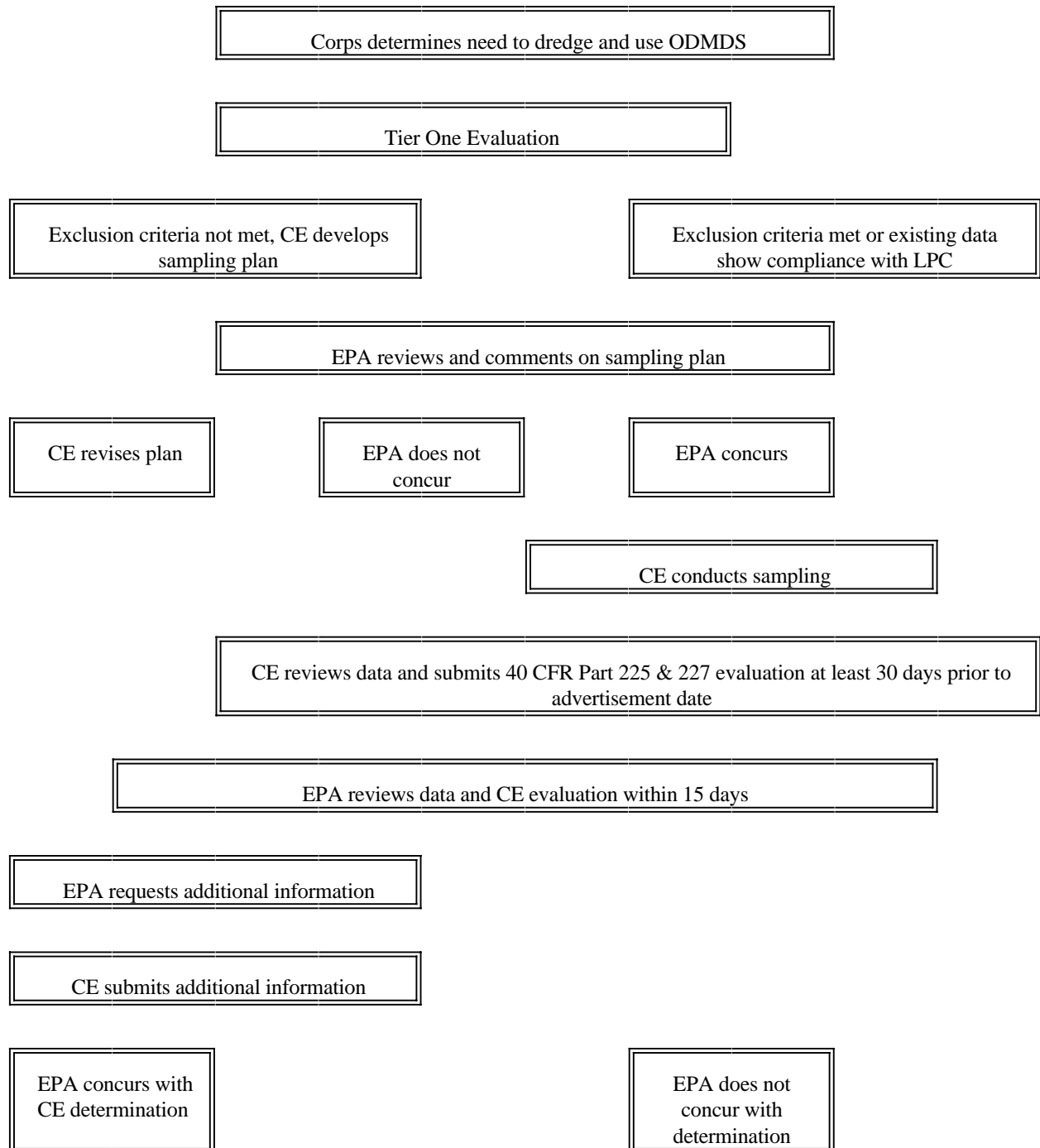
### **2.2 Exclusionary Criteria, Need for Testing and Testing Plan Development Determinations**

Available information will be evaluated early in the review of proposed dredging projects, by both the CE District and EPA Region IV, to determine whether the dredged material needs testing and, if so, how. Appendix B (Section 1, 2, and part of 3) describes the information that will be used by the CE District and EPA Region IV to make these decisions and avoid delays in project implementation.

Information on the proposed dredging site, sediment grain size, and potential for contamination is used to determine whether the exclusion criteria are met (40 CFR 227.13 (b)). Core boring logs, dredging design specifications, area hydrology, and locations, quantities, history, and types of pollutants discharged upstream of the proposed dredging are used for this determination. If the criteria are not met, additional information on previous testing (results and dates) and dredging (dates and extent of dredging) are used to determine the testing needs.

Should testing be required, the previously mentioned information will also be used in development of a sampling and testing plan. This plan will include mutually agreed upon contaminants of concern, method detection limits, test organisms, number and location of samples, sampling procedure and other plan components.

**Figure 2.1. Information submittal and review process.**



Timely decision making is critical to avoid delays in project implementation, especially for the preliminary determinations. The time frame to complete an assessment of the need for testing, developing a test plan, collecting and analyzing samples, running biotoxicity and bioaccumulation tests, performing appropriate statistical analyses and preparing the sampling and testing report could take eight or more months. To complete required evaluations, the process should be started at least six months prior to the proposed dredging. If the project is likely to be more complex, additional time should be allowed.

### **3.0 ADMINISTRATIVE PERMIT REQUIREMENTS**

#### **3.1 MPRSA Section 103 Permit Requirements**

Applications for MPRSA Section 103 permits for the transportation of dredged material, for the purpose of disposal at an approved Ocean Dredged Material Disposal Site (ODMDS), must be submitted to the CE District office (see Figure 2.1 for an outline of Section 103 permit procedures). MPRSA Section 103 applications must comply with CE permitting regulations in 33 CFR Parts 320 to 330, and 335 to 338. All information submitted as part of the MPRSA application process, including Civil Works Projects, must also comply with EPA Ocean Dumping Regulations in 40 CFR Parts 220 to 228.

The CE District will coordinate all sediment testing plans with EPA Region IV. Pre-application conferences to prepare appropriate sampling plans are encouraged for all MPRSA Section 103 permit applicants. The CE District is responsible for coordination of all federal actions, including EPA concurrences, pertaining to MPRSA Section 103 applications. The applicant may also need to coordinate activities with the appropriate state regulatory agencies for compliance with State Water Quality Certification (Clean Water Act Section 401), and the State Coastal Zone Consistency [Coastal Zone Management Act Section 307(c)].

The following information will be required when applying for a MPRSA Section 103 permit or planning a civil works project:

1. The proposed type of dredging, disposal, and navigation equipment to be used.
2. An estimate of the total amount of dredged material to be excavated from the proposed site, including channel dimensions.
3. An evaluation of dredged material disposal alternatives including an examination of potential beneficial uses of the proposed dredged material and a consideration of alternative disposal options before selecting the ocean disposal option (40 CFR Sections 227.14 to 227.16). Documentation of the criteria used as the basis upon which selections or rejections were made. If prior evaluations are current, reference to them is encouraged.
4. Written documentation of the site dredging history, including all results from previous sediment testing (both abiotic and biotic) and a general survey of other prior or current dredging activities at or near the site. If prior evaluations are current, reference to them is encouraged.
5. If the ocean disposal application for re-certification of the proposed dredged material is currently covered under a MPRSA Section 103 maintenance dredging permit, the permit number (or Public Notice and date) should be provided. If more than three years has passed since the last evaluation was conducted for the dredge site, or data are considered to be inadequate, then the CE District will

evaluate the need, in consultation with EPA Region IV, for additional evaluation.

6. Detailed information, along with written documentation, on known or suspected site contamination including oil, chemical, or waste spills and any other discharges that may cause contamination of the proposed dredging site. The local U.S. Coast Guard and Port Authority offices should be contacted to obtain additional information on spills or suspected contamination. Any chemicals known or suspected of contaminating the proposed dredging site must be added to the list of possible contaminants of concern in Section 6.0 of this manual.

### **3.2 Corps of Engineers Public Notices**

Once the CE District receives a completed permit application, the information will be published for review as a Public Notice. In addition to information required by the CE District, the Public Notice must contain the following information as specified in 40 CFR Section 225.2 (a):

1. The location (latitude and longitude) of the proposed disposal site boundaries;
2. A statement about whether the disposal site has been designated pursuant to MPRSA Section 102(c);
3. If the proposed disposal site has not been designated by EPA, a statement of the basis for the proposed determination describing why no previously designated site is feasible and a description of the characteristics of the proposed disposal site will be necessary for designation as a MPRSA Section 103 site by the CE District Engineer. The Public Notice for MPRSA Section 103 site must comply with applicable sections of 40 CFR Part 228;
4. Known historical uses of the proposed disposal site;
5. Existence and documented effects of other authorized placement at the disposal site;
6. An estimate of the length of time required for dredged material disposal at the disposal site;
7. Characteristics and composition of the proposed dredged material; including information relative to the status of physical, chemical and biological tests on the proposed dredged material;
8. A statement of need for an environmental impact statement.

### **3.3 Determination of Compliance with EPA's Ocean Dumping Criteria**

Information provided in the Public Notice and other pertinent information will be used by the CE District to aid in determining the suitability of the proposed dredged material for ocean disposal under the criteria defined in 40 CFR Part 227 (see Appendix B for Section 103 Evaluation Report). If the data submitted by the applicant are insufficient to evaluate the proposed dredged material and prepare the Section 103 Evaluation Report (Appendix B) the CE, with the cooperation of EPA Region IV, will request additional information [40 CFR Section 225.2(b)]. The CE District will furnish the Section 103 Evaluation Report to EPA Region IV for independent review and concurrence relative to the suitability of the dredged material for ocean disposal [40 CFR 225.2(c)]. EPA Region IV will inform the appropriate CE District in writing as to their determination within 15 days of receipt of the Section 103 Evaluation Report. The EPA Regional Administrator may, however, request an extension of this 15 day period to 30 days. If the EPA finds that the material does not meet the EPA Ocean Dumping Criteria then the CE can not issue the permit unless procedures for invoking an economic impact waiver are initiated by the CE District Engineer [40 CFR Sections 225.2(e), 225.3 and 225.4].

## **4.0 TIERED TESTING AND THE 1991 GREEN BOOK**

EPA and the CE have developed a tiered testing approach to evaluate the suitability of dredged material for ocean disposal (see Figure 4.1 for an outline of the Tiered Testing Approach). This approach is defined in detail in Chapters 1-4 of the 1991 Green Book (EPA and CE, 1991). A brief description of tiered testing is presented below for use in developing adequate sampling and testing plans.

### **4.1 Tier I - A Comprehensive Review of Existing Information**

The purpose of Tier I is to determine if a decision on compliance with the limiting permissible concentration (LPC) can be made using existing information. The LPC, defined in 40 CFR Section 227.27, is based on initial mixing, marine water quality criteria, and results from the toxicity and bioaccumulation testing of the proposed dredged material and reference sediment. Tier I is a comprehensive analysis of all existing and readily available information pertinent to the proposed dredging project, including all previously collected physical, chemical and biological data. Tier I evaluations begin with a comparison of existing physical and chemical information on the proposed dredged material with the three exclusion criteria of 40 CFR Section 227.13(b). If the dredged material meets at least one of these criteria, additional testing is not required.

The three exclusion criteria are:

(1) The dredged material is composed primarily of sand, gravel, rock, or any other naturally occurring bottom material with particle sizes larger than silt, and the material is found in areas of high current or wave energy such as streams with large bed loads or coastal areas with shifting bars and channels; or

(2) The dredged material is for beach nourishment or restoration and is composed primarily of sand, gravel, or shell with particle sizes compatible with material on the receiving beach; or

(3) When:

(a) the material proposed for disposal is substantially the same as the substrate at the proposed dump site; and

(b) the site from which the material proposed for disposal is to be taken is far removed from known sources of pollution so as to provide a reasonable assurance that such material has not been contaminated by such pollution.



Figure 4.1 Unavailable

Conclusive written documentation must be provided showing that the proposed material meets one of the exclusion criteria. If the proposed dredged material does not meet any of the exclusion criteria, sediment characterization data can be used to show that the LPC of the sediment can be met. If one or more of the exclusionary criteria are then satisfied further evaluation of the dredged material will be unnecessary. A critical component of the Tier I evaluation is deciding which, if any, contaminants of concern are present in the dredged material (see Table 6.2). The contaminants of concern must be identified on a case by case basis. In identifying possible contaminants, those chemicals necessary to determine compliance with the requirements of Part 227.6 of the regulations must be included. Other possible contaminants that should be included are those that might be expected to cause unacceptable adverse impacts, if placed in the ocean. In some dredged materials, there may be no contaminants of concern. The contaminants of concern in the dredged material should be identified based on:

1. Presence in the dredged material;
2. Presence in the dredged material relative to the concentration in the reference material;
3. Toxicological importance;
4. Propensity to bioaccumulate from sediments.

Sources of potential information for a Tier I evaluation include the following:

1. Results from prior physical, chemical, and biological tests of the proposed material to be disposed;
2. Results of prior field monitoring studies of the material proposed to be dumped;
3. Information describing the source of the material to be disposed in the ocean which would be relevant to the identification of potential contaminants of concern;
4. Existing data contained in other EPA or CE files or are otherwise available from public or private sources. Examples include the following:
  - a. Selected Chemical Spill Listing (EPA);
  - b. Pesticide Spill Reporting System (EPA);
  - c. Pollution Incident Reporting System (U.S. Coast Guard);
  - d. Identification of In-Place Pollutants and Priorities for Removal (EPA);

- e. Hazardous waste sites and management facilities reports (EPA);
- f. CE studies of sediment pollution and sediments;
- g. Federal STORET, BIOS, and ODES databases (EPA);
- h. Water and sediment data on major tributaries (Geological Survey);
- i. NPDES permit records;
- j. CWA 404(b)(1) Evaluations;
- k. Pertinent and applicable research reports;
- l. MPRSA 103 Evaluations;
- m. Port Authorities;
- n. Colleges/Universities;
- o. State environmental agencies;
- p. Published scientific literature.

In some cases, it may be necessary to supplement available information with more recent physical and chemical analyses of the proposed dredged material. Additional tests for chemicals of concern include compounds known or suspected of contaminating the dredging site which may include those on the list of compounds found in Section IV of this manual. Water chemistry is not routinely required; however the CE may require water chemistry analyses based on specific projects. If adequate information is not available for Tier I compliance, the evaluation advances to Tier II.

#### **4.2 Tier II - Water - Column and Theoretical Benthic Impact Analyses**

Tier II evaluations consist of a determination of compliance with applicable water quality criteria (WQC) using a numerical mixing model of disposal site conditions, and an evaluation of benthic impact using calculations to determine the Theoretical Bioaccumulation Potential (TBP) for chemical contaminants of concern (EPA and CE 1991, see page 5-1). The purpose of Tier II is to provide a reliable, rapid screen for potential adverse impacts, thereby limiting subsequent testing. The following criteria will need to be satisfied in order to determine compliance in Tier II:

1. The ocean disposal of dredged material can not exceed applicable EPA, or state if applicable, WQC outside the disposal site boundaries at any time or within the disposal site boundaries 4 hours

after of initial mixing. The EPA WQC are listed in Table 4.2. The Tier II WQC evaluation can only be bypassed if there are no current WQC for the contaminants of concern (EPA and CE 1991, page 5-2). The following steps maybe included in a Tier II evaluation:

- a. Tier II water column evaluations are conducted using the numerical model supplied in Appendix B of the 1991 Green Book. This model is a screening tool which assumes that all contaminants are released into the water column during the disposal process;
  - b. If additional water column testing is necessary, after modelling data are reviewed, elutriate tests must be performed as described in Chapters 9 and 10 of the 1991 Green Book;
  - c. If WQC have not been established for all of the chemicals of concern found in the proposed dredged material or if synergistic effects are expected, further testing in Tier III will be required to determine if compliance with the LPC will be met (EPA and CE 1991, pages 5-3 to 5-4).
2. At present only the TBP of nonpolar organic chemicals, such as polychlorinated biphenyls (PCB's), and pesticides, can be determined from dredged material samples in Tier II. The evaluation of the TBP is based on the concentration of nonpolar organic chemicals, total organic carbon in the sediment, and lipid concentrations in benthic organisms. The TBP predicts the magnitude of bioaccumulation likely to occur with nonpolar organic chemicals found in the proposed dredged material (EPA and CE 1991, page 5-1).
- a. Guidance for calculating the TBP of nonpolar organic chemicals is provided in Chapter 10 of the 1991 Green Book.
  - b. If polar organic chemicals, organometals or trace metals are considered to be contaminants of concern in the proposed dredged material, further testing will be required in Tier III or Tier IV (EPA and CE 1991, page 5-4).

**Table 4.2 EPA Water Quality Criteria<sup>1</sup> (WQC) for Chemicals of Concern in Marine Waters.**

<b>Chemicals of Concern</b>	<b>Acute Concentration Levels (Tg/l)<sup>2</sup></b>	<b>Chronic Concentration Level (Tg/l)<sup>2</sup></b>
<b>Metals</b>		
Cadmium	43	9.3
Chromium (III)	1030	103
Chromium (VI)	1100	50
Copper	2.9	2.9
Lead	140	5.6
Mercury	2.1	0.0251
Nickel	75	8.3
Selenium	300	71
Silver	2.3	NA
Thallium	213	21.3
Zinc	95	86
<b>Nonmetals</b>		
Ammonia	233	35
Arsenic	69	35
Cyanide	1	1
<b>Pesticides</b>		
Aldrin	1.3	0.13
Chlordane	0.09	0.004
DDT	0.13	0.001
DDE	1.4	0.14

**Table 4.2 EPA Water Quality Criteria<sup>1</sup> (WQC) for Chemicals of Concern in Marine Waters.**

<b>Chemicals of Concern</b>	<b>Acute Concentration Levels (Tg/l)<sup>2</sup></b>	<b>Chronic Concentration Level (Tg/l)<sup>2</sup></b>
DDD	0.25	0.25
Dieldrin	0.71	0.0019
I,J - Endosulfan	0.034	0.0087
Endrin	0.037	0.0023
Heptachlor	0.053	0.0036
Heptachlor Epoxide	0.053	0.0036
γ-Hexachlorocyclohexane (Lindane)	0.016	NA
Toxaphene	0.21	0.0002
<b>Organic Compounds</b>		
<b>Phenol and Substituted Phenols</b>		
Phenol	580	58
2,3,5,6- Tetrachlorophenol	NA	440*
Pentachlorophenol	13	7.9
Nitrophenols	4,850*	NA
4-Nitrophenol	717	71.7
2,4,-Dinitrophenol	285	48.5
<b>Phthalate Esters</b>		
Total	2,944*	3.4*
Butylbenzyl Phthalate	294.4	29.4
Diethyl Phthalate	759	75.9
Dimethyl Phthalate	5800	580
Di-n-Butyl Phthalate	NA	3.4

<b>Table 4.2 EPA Water Quality Criteria<sup>1</sup> (WQC) for Chemicals of Concern in Marine Waters.</b>		
<b>Chemicals of Concern</b>	<b>Acute Concentration Levels (Tg/l)<sup>2</sup></b>	<b>Chronic Concentration Level (Tg/l)<sup>2</sup></b>
<b>Polychlorinated Biphenyls (PCB's)<sup>3</sup></b>		
PCB-1016	1.05	0.03
PCB-1221	" "	" "
PCB-1232	1.05	0.03
PCB-1242	" "	" "
PCB-1248	" "	" "
PCB-1254	" "	" "
PCB-1260	" "	" "
Total PCB's	10	" "
<b>Polynuclear Aromatic Hydrocarbons (PAH's)</b>		
Total PAH's	300*	NA
Anthracene	97	9.7
Fluoranthene	4	1.6
Naphthalene	235	23.5

<sup>1</sup> Reference: U.S. Environmental Protection Agency (1987e).

<sup>2</sup> Concentrations in  $\mu\text{g/l}$  unless otherwise stated

<sup>3</sup> Analysis of PCB Aroclors are no longer required. The 1991 Green Book however requires testing for PCB congeners using methods defined in Tetra Tech (1986a) and NOAA (1989).

NA = Not available.

\* = Insufficient data to develop criteria. Value shown is the Lowest Observed Effect Level (LOEL).

#### **4.3 Tier III - Bioassay and Bioaccumulation Tests of Proposed Dredged Material**

If the Tier I and/or Tier II evaluations raise concerns about contamination in the proposed dredged material or provide information to make a decision regarding the acceptability of the dredged material for disposal in the ocean then bioassay and bioaccumulation tests will be required. Tier III provides guidance on how to assess the effects of dredged material on appropriately sensitive marine

organisms. Guidance for these tests is discussed on pages 11-1 to 11-17 of the 1991 Green Book. Lists of appropriately sensitive marine species can be found in Tables 7.1, 7.2 and 7.3 of this manual.

Tier III procedures include bioassay and bioaccumulation tests on water column and benthic organisms. Results from these tests are usually sufficient to evaluate the suitability of the proposed dredged material for ocean disposal. In rare cases, Tier III testing may indicate that additional toxicity and/or bioaccumulation testing will be required in accordance with Tier IV guidance (EPA and CE 1991, page 6-1). The components of a Tier III evaluation may include the following:

1. Chemical, bioassay and bioaccumulation testing if the presence of contaminants of concern in the proposed dredged material is suspected [40 CFR Section 227.32];

2. Bulk sediment chemistry tests to refine the chemicals of concern for subsequent biological testing and to aid in the interpretation of test results;

3. Water-column bioassay tests to assess the effects of the proposed dredged material on pelagic organisms;

- a. Water - column bioassays may be used if there are no applicable marine WQC for the contaminants of concern, or synergistic effects between certain contaminants are suspected.

- b. Water - column bioassays are evaluated using the initial mixing and LPC of the material. Results of the 100 percent concentration test are also used as a tool to determine if the undiluted particulate phase impacts test species, before the initial mixing and LPC calculations are made.

4. Whole - sediment bioassays to evaluate the effects of the proposed dredged material on benthic organisms;

- a. All whole - sediment compliance evaluations use mortality data from the whole sediment treatments. A dilution series similar to the suspended phase tests is not used.

- b. Proposed dredged material does not meet the ocean dumping criteria for the whole sediment bioassay when mortality:

- (i). Is statistically significantly higher in the dredged material tests than the reference sediment tests, and;

- (ii). Exceeds the reference sediment mortality by at least 10 percent, or;

- (iii). Exceeds the reference sediment mortality by at least 20 percent for .  
the 10 day amphipod whole - sediment bioassay test (EPA and CE 1991, page 6-2).



5. Bioaccumulation tests evaluate the bioavailability of contaminants of concern in the proposed dredged material. Guidance on bioaccumulation testing is provided in Chapter 12 of the 1991 Green Book.

- a. Bioaccumulation tests are conducted for 10 days if heavy metals contamination is suspected, or 28 days if organic chemical contamination is suspected.
- b. If both types of contamination are suspected, then bioaccumulation tests are conducted for 28 days.
- c. The contaminant concentrations in the tissues of the test species are compared with:
  - (i). The Food and Drug Administration (FDA) published list of Action Levels for Poisonous or Deleterious Substances in Fish and Shellfish for Human Food of 1991 (see EPA and CE 1991, page 6-5). If the contaminants of concern exceed the FDA Action Limits, the dredged material will be unsuitable for ocean disposal.
  - (ii). Contaminant tissue concentrations that do not exceed the FDA Action Limits are also statistically compared to tissue concentrations from test species exposed to reference sediments. If the concentrations of the contaminants of concern exceed those in the reference sediments then evaluations of LPC compliance for the proposed dredge material will be made on a case by case basis.

The following factors will be used to evaluate LPC compliance when bioaccumulation of contaminants in dredged material statistically exceeds those in the reference sediment. The factors and their order of evaluation are as follows:

Factors 1 - 4 will be evaluated first by the CE and EPA and are:

1. Magnitude by which bioaccumulation in the dredged material exceeds bioaccumulation in the reference material;
2. Number of contaminants for which bioaccumulation in the dredged material is statistically greater than bioaccumulation in the reference material;
3. Number of species in which bioaccumulation in the dredged material is statistically greater than bioaccumulation in the reference material; and
4. Toxicological importance of the contaminants whose bioaccumulation in the dredged material statistically exceeds that of the reference material.

If a compliance decision cannot be agreed to after reviewing factors 1 - 4, factors 5 - 7 will be evaluated. Factors 5 - 7 include:

5. Phylogenetic diversity of the species in which bioaccumulation in the dredged material statistically exceeds bioaccumulation in the reference material;
6. Propensity for the contaminants with statistically significant bioaccumulation to biomagnify within aquatic food webs; and
7. Magnitude of toxicity and number and phylogenetic diversity of species exhibiting greater mortality in the dredged material than in the reference material.

If a compliance decision still cannot be reached, a sampling plan will be developed and agreed upon by both the EPA and the CE to evaluate factor 8.

Factor 8 is the magnitude by which contaminants whose bioaccumulation in the dredged material exceeds that in the reference material and also exceeds the concentrations found in comparable species living in the vicinity of the proposed disposal site.

Based on this tiered evaluation, the CE District will determine if the proposed dredged material is suitable for ocean disposal. EPA Region IV will perform an evaluation of these data to independently determine the suitability of the proposed dredge material for ocean disposal.

#### **4.4 Tier IV - Case Specific Testing**

Under certain circumstances, it may be necessary to evaluate the long-term effects of the proposed dredged material on appropriately sensitive marine organisms. In this case the appropriate CE District, in consultation with EPA Region IV, will determine the required tests to evaluate suspected chronic or other effects. Close coordination with the Federal regulatory agencies, including EPA Office of Research and Development and the CE Waterways Experiment Station, are required for Tier IV testing.

## **5.0 SEDIMENT SAMPLING**

### **5.1 Selection of Sampling Stations**

The selection of sampling stations at the proposed dredging site and the collection of sediment at reference and control stations are critical steps in designing an acceptable sediment sampling plan. The CE District, with EPA review and approval, will design a sampling plan using guidance provided in Chapter 8 of the 1991 Green Book. Table 5.1 details specific factors to be considered when designing sediment sampling plans. In addition to this guidance, the sampling plan will consider the following factors:

1. The volume of sediment to be dredged, the areal extent of dredging, the depth of sediment to be dredged, and the heterogeneity of the sediment at the proposed dredging site;
2. The known physical, chemical, and toxicological characteristics of the proposed dredging site;
3. Hydrological and depositional characteristics of the dredging site;
4. Pollution sources; and
5. Statistical Power.

### **5.2 Sampling Reference Stations**

If the proposed dredged material does not meet the testing exclusion criteria defined in 40 CFR Section 227.13(b), physical, chemical, bioassay, and bioaccumulation tests may be required. The test results from proposed dredging site samples are compared to test results from appropriate reference site sediments. Reference sediment is defined as: "A sediment that is: (a) substantially free of contaminants; (b) similar in grain size, as practicable, to that of the disposal site; and (c) reflects conditions that would exist in the vicinity of the disposal site if no dredged material disposal had ever occurred, but all other influences on sediment had taken place." Reference sediment sampling stations will be selected, by the CE District in consultation with EPA, to simulate conditions at the proposed disposal site in the absence of past dredged material disposal. Test organisms will be selected that are not sensitive to possible sediment grain size differences among the reference site, the control site and the proposed dredging site.

Reference sediments may be collected from: (1) a single reference-sediment sampling location (known as the reference-point approach); or (2) from a number of locations within a reference area (known as the reference-area approach). In the reference area approach, the reference location is not viewed as a single station or point but as a collection of sediment samples from the entire reference area, excluding the disposal site itself. Reference samples may be composited and tested according

to guidance provided in Chapter 8 of the 1991 Green Book.

Replicate sediment samples should be collected at the reference site using an appropriate collection device (see Table 5.1). Replicates may be composited into a single sample then press sieved to remove large particles. The collected sediment should be of sufficient quantity to conduct all required testing. A minimum of three replicate sediment samples from the reference site will be required for all testing. The CE District, with EPA approval, will determine appropriate sampling devices, techniques and location for reference samples. EPA and CE Districts agree that efforts will be made to identify reference areas acceptable to both EPA and CE Districts that will be used for multiple disposal sites. These reference samples may be used for up to five years with agreement from EPA Region IV and the CE District.

Specific reference sites have been identified within the boundaries of the Mobile District. These sites are identified in Appendix E. As other CE districts in SAD identify additional reference sites, these will be added to Appendix E.

<b>Table 5.1. Summary of recommended procedures for sample collection, preservation and storage<sup>a</sup></b>						
Analysis or Test Duration	Collection Method	Amount Required <sup>*</sup>	Container <sup>**</sup>	Preservation Technique	Storage Container	Storage <sup>***</sup>
<b>SEDIMENT</b>						
Chemical/Physical Analysis						
Bulk Metals	Grab/corer with lexan liner	100 g	Precleaned polyethylene jar <sup>c</sup>	Dry ice <sup>c</sup>	≤20°C	Hg - 30 days Others - six months <sup>h</sup>
Bulk Organics (PCB's, Pesticides, High Molecular Weight (HMW) Hydrocarbons)	Grab/corer	250 g	Solvent-rinsed glass jar with Teflon Lid <sup>c</sup>	Dry ice <sup>c</sup>	≤20°C/dark <sup>d</sup>	10 days <sup>d</sup>
Particle Size	Grab/corer	100 g	Whirl-Pac <sup>c</sup>	Refrigerate	< 4°C	Undetermined
TOC	Grab/corer	50 g	Heat treated glass vial with Teflon-lined lid	Dry ice <sup>c</sup>	< 4°C/dark	Undetermined
Total Solids/ Specific Gravity	Grab/corer	50 g	Whirl-Pac <sup>c</sup>	Refrigerate	< 4°C	Undetermined
Miscellaneous	Grab/corer	≥ 50 g	Whirl-Pacc	Refrigerate	< 4°C	Undetermined
Sediments used for elutriate testing	Grab/corer	1 l	Glass with Teflon-lined lid	Completely fill and Refrigerate	< 4°C/dark/airtight	Undetermined

<b>Table 5.1. Summary of recommended procedures for sample collection, preservation and storage<sup>a</sup></b>						
Analysis or Test Duration	Collection Method	Amount Required*	Container**	Preservation Technique	Storage Container	Storage***
Biological Testing						
Dredged Material	Grab corer	12-15 l/sample	Plastic bag or contained <sup>c</sup>	Completely fill and Refrigerate	4°C/dark/airtight	2 weeks <sup>f</sup>
Reference Sediment	Grab/corer	45-50 l/test	Plastic bag or contained <sup>c</sup>	Completely fill and Refrigerate	4°C/dark/airtight	2 weeks <sup>f</sup>
Control Sediment	Grab/corer	21-25 l/test	Plastic bag or contained <sup>c</sup>	Completely fill and Refrigerate	4°C/dark/airtight	2 weeks <sup>f</sup>
<b>WATER AND ELUTRIATE</b>						
Particulate Analysis	Discrete sampler or pump	500-2000 ml	Plastic or glass <sup>b</sup>	Lugols solution and refrigerate	4°C	Undetermined
Metals	Discrete sampler or pump	1 l	Acid-rinsed polyethylene or glass job <sup>g</sup>	pH<2 with HNO <sub>3</sub> <sup>g</sup>	4°C 2°C <sup>g</sup>	Hg - 30 days Others - six months <sup>h</sup>
Total Kjeldahl Nitrogen (TKN)	Discrete sampler or pump	100-200 ml	Plastic or glass <sup>h</sup>	pH<2 with HNO <sub>3</sub> <sup>g</sup> ; refrigerate	4°C <sup>h</sup>	24 hr <sup>h</sup>
Chemical Oxygen Demand (COD)	Discrete sampler or pump	200 ml	Plastic or glass <sup>h</sup>	pH<2 with HNO <sub>3</sub> <sup>g</sup> ; refrigerate	< 4°C <sup>h</sup>	7 days <sup>h</sup>
Total Organic Carbon (TOC)	Discrete sampler or pump	100 ml	Plastic or glass <sup>h</sup>	pH<2 with HNO <sub>3</sub> <sup>g</sup> ; refrigerate	< 4°C <sup>h</sup>	< 48 hrs <sup>h</sup>

<b>Table 5.1. Summary of recommended procedures for sample collection, preservation and storage<sup>a</sup></b>						
Analysis or Test Duration	Collection Method	Amount Required <sup>*</sup>	Container <sup>**</sup>	Preservation Technique	Storage Container	Storage <sup>***</sup>
Total Inorganic Carbon (TIC)	Discrete sampler or pump	100 ml	Plastic or glass <sup>h</sup>	Airtight seal; refrigerate <sup>h</sup>	< 4°C <sup>h</sup>	6 months <sup>h</sup>
Phenolics	Discrete sampler or pump	1 l	Glass <sup>h</sup>	0.1-1.0 g CuSO <sub>4</sub> ; H <sub>2</sub> SO <sub>4</sub> to pH <2; refrigerate	< 4°C <sup>h</sup>	24 hr <sup>h</sup>
Soluble Reactive Phosphates	Discrete sampler or pump	--	Plastic or glass <sup>h</sup>	Filter and refrigerate <sup>h</sup>	< 4°C <sup>h</sup>	24 hr <sup>h</sup>
Organics	Discrete sampler or pump	4 l	Amber glass bottle <sup>g</sup>	Airtight seal; refrigerate <sup>h</sup>	< 4°C 2°C <sup>g</sup>	5 days <sup>g</sup>
Volatile Organics	Discrete sampler or pump	80 ml	Glass vial <sup>g</sup>	ph < 2 with 1:1 HCL in airtight completely filled container <sup>g</sup>	< 4°C 2°C <sup>g</sup>	5 days <sup>g</sup>
Total Phosphorus	Discrete sampler or pump	--	Plastic or glass <sup>h</sup>	Refrigerate	4°C <sup>h</sup>	7 days <sup>h</sup>
Total Solids	Discrete sampler or pump	200 ml	Plastic or glass <sup>h</sup>	Refrigerate	4°C <sup>h</sup>	7 days <sup>h</sup>
Volatile Solids	Discrete sampler or pump	200 ml	Plastic or glass <sup>h</sup>	Refrigerate	4°C <sup>h</sup>	7 days <sup>h</sup>
Sulfides	Discrete sampler or pump	--	Plastic or glass <sup>h</sup>	2 ml ZnOAc <sup>h</sup>	Ambient <sup>h</sup>	24 hrs <sup>h</sup>

Table 5.1. Summary of recommended procedures for sample collection, preservation and storage <sup>a</sup>						
Analysis or Test Duration	Collection Method	Amount Required*	Container**	Preservation Technique	Storage Container	Storage***
<b>Tissue</b>						
Trace Metals	Trawl/Teflon coated grab	5-10 g	Double Ziploc <sup>c</sup>	Handle w/nonmetallic forceps; plastic gloves; dry ice <sup>c</sup>	≤20°C <sup>c</sup>	Hg - 28 days Others- six months <sup>i</sup>
PCB's and Chlorinated Pesticides	Trawl/Teflon coated grab	10-25 g	Hexane-rinsed double aluminum foil and double Ziploc <sup>c</sup>	Handle w/hexane rinsed stainless steel forceps; dry ice <sup>c</sup>	≤20°C <sup>c</sup>	10 days <sup>i</sup>
Volatile Organics	Trawl/Teflon coated grab	10-25 g	Heat-cleaned aluminum foil and watertight plastic bag <sup>i</sup>	Covered ice chest <sup>d</sup>	≤20°C <sup>c</sup>	10 days <sup>i</sup>
PAH's	Trawl/Teflon coated grab	10-25 g	Hexane-rinsed double aluminum foil and double Ziploc <sup>c</sup>	Handle w/hexane rinsed stainless steel forceps; dry ice <sup>c</sup>	≤20°C <sup>c</sup>	10 days <sup>i</sup>
Lipids	Trawl/Teflon coated grab	part of organic analyses	Hexane-rinsed double aluminum foil <sup>c</sup>	Handle w/hexane rinsed stainless steel forceps; quick freeze <sup>c</sup>	≤20°C <sup>c</sup>	Undetermined



- a This table contains only a summary of collection, preservation, and storage procedures for samples. The cited references should be consulted for a more detailed description of these procedures.
- b These are holding times for sediment, water, and tissue. References should be consulted if holding times for sample extracts are desired.
- c NOAA 1989
- d Tetra Tech 1986a
- e Polypropylene should be used if phthalate bioaccumulation is of concern.
- f Two weeks is recommended; sediments must not be held for more than 6 weeks prior to biological testing.
- g EPA (1987); 40 CFR Part 136; Table III
- h Plumb 1981
- i Tetra Tech 1986b

\* Amount required is that delivered to the laboratory. Wet weight or volume provided as appropriate. Miscellaneous sample size for sediment should be increased if auxiliary analytes that can not be included as part of the organic or metal analyses are added to the list. The amounts shown are not intended as firm values; more or less tissue may be required depending on the analytes, matrices, detection limits and particular analytical laboratory.

\*\* All containers should be certified as clean according to EPA (1990).

\*\*\* Holding times are from the time of sample collection.

### **5.3 Sampling Control Site Stations**

Control sediment must be used in all bioassay and bioaccumulation tests. Control sediment is distinguished from the reference sediment because it is selected to provide optimum conditions for the organisms. The control samples are used to determine the general health of the test organisms during the bioassay and bioaccumulation tests, and to evaluate test protocols as part of the laboratory QA/QC program. The control sediment should also be press sieved to remove large particles and biological material. The coordinates of the control site must be documented in a sampling plan and approved by the appropriate CE District and EPA Region IV prior to collection.

1. Control sediment shall be defined as: "A natural sediment essentially free of contaminants and compatible with the biological needs of the test organisms such that the sediment has no discernible influences on responses being measures in the tests" (EPA and CE 1991, page 1-5).
2. Control sediment is used in the water-column and whole-sediment bioassay tests to assess the overall health of the test species. With the exception of the 10 day whole sediment test, the average control test species mortality should not exceed 10 percent. In the case of whole sediment testing, the average control mortality should not exceed 20 percent. In the event that these levels are exceeded, testing may need to be repeated.
3. When bioaccumulation testing requires the depuration of sensitive species (i.e., those species that can not be depurated solely in clean water for periods of 24 to 48 hours) prior to analysis, test species should be depurated in control sediments. The test results will be used to determine the levels that chemicals of concern bioaccumulated in the animals depurated in the control sediments.
4. The control sediment tests are not usually compared to the proposed dredged material as part of the analysis to determine whether sediments are suitable for ocean disposal.

### **5.4 Sampling the Proposed Dredging Site**

Sediment sampling, at the selected stations in the proposed dredging site, should be designed to ensure that the proposed dredged material will be adequately characterized. This sampling should include consideration of project design, the dredging history of the area (i.e., new vs. maintenance work), sedimentation rates, and any previous sampling. Sample collection methods (i.e., grab, dredge, coring) can have a effect on sediment integrity. Therefore, it is important to understand the advantages and disadvantages of each sampling device for the type of testing that is to be done (ASTM 1990a). Sediment sampling documentation should include:

1. A description of the amount and extent of the proposed dredging as well as other factors previously described in Section 5.1. Sample location positioning should be precise to  $\pm 10$  meters will be required;

2. The amount of sediment to be collected to perform all physical, chemical, bioassay and bioaccumulation sediment testing. Consideration of acceptable storage and holding times should be given depending on the test to be conducted (EPA and CE, 1991, page 8-15); and

3. Sample logs requirements which will document sediment sample handling procedures. Sample logs must specifically include: (a) sample date; (b) the sample location (latitude and longitude); (c) sample identification code for chain of custody documentation, description of sediment odor and physical appearance; (d) sample depth and water depth; (e) sampling method (including sampling gear); (f) and number of samples taken; (g) any problems encountered.

## 6.0 PHYSICAL AND CHEMICAL TESTING OF DREDGED MATERIAL

Strict adherence to established testing protocols and detection limits while conducting sediment physical (Table 6.0) and chemical analyses (Table 6.1) is required. Any deviation from these protocols or detection limits must be acceptable to the CE District and EPA Region IV, prior to analysis. All data should be reported as dry weight unless otherwise specified. Established QA/QC procedures must be followed (see Section 9.0). In all cases, CE or EPA personnel will inspect testing laboratories.

<b>Table 6.0. Sediment Analyses.</b>		
<b>Parameter</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limits</b>
Percent Solids	Plumb 1981	1.0% solids
Grain Size Distribution	" "	1.0% <sup>2</sup>
Total Organic Carbon	9060	0.1%

<sup>1</sup> EPA unless otherwise noted

<sup>2</sup> Grain Size tests results must be reported on ENG Form 2087.

<b>Table 6.1. Possible Chemicals to be Analyzed from Sediment Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit</b>
<b>Metals</b>		
Antimony	7040, 7041	0.50 mg/kg
Arsenic	7060, 7061	" "
Beryllium	7090, 7091	0.10 mg/kg
Cadmium	7130, 7131	" "
Chromium	7190, 7191	" "
Copper	7210, 7211	" "
Lead	7420, 7421	" "
Mercury	7471	0.05 mg/kg
Nickel	7520, 7521	0.10 mg/kg
Selenium	7740, 7741	0.20 mg/kg

<b>Table 6.1. Possible Chemicals to be Analyzed from Sediment Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit</b>
Silver	7760, 7761	0.10 mg/kg
Thallium	7840, 7841	" "
Zinc	7950, 7951	0.01 mg/kg
<b>Nonmetals</b>		
Ammonia	Plumb 1981	0.10 mg/kg
Cyanide	9010, 9012	1.0 mg/kg
2,3,7,8- TCDD, TCDF & Congeners	8290	1 ng/kg
Total Sulfides	9030, Plumb 1981	0.20 mg/kg
Total Organic Carbon	9060	0.1%
<b>Pesticides</b>		
Aldrin	8080	0.01 mg/kg
Chlordane & Derivatives	" "	" "
Dieldrin	" "	" "
DDT & Derivatives	" "	" "
Endosulfan & Derivatives	" "	0.02 mg/kg
Endrin & Derivatives	" "	0.01 mg/kg
Heptachlor & Derivatives	" "	0.02 mg/kg
Hexachlorocyclohexane & Derivatives	" "	0.01 mg/kg
Methoxychlor	" "	0.02 mg/kg
Toxaphene	" "	0.01 mg/kg
<b>Organic Compounds</b>		
<b>Polynuclear Aromatic Hydrocarbons (PAH's)</b>		
Acenaphthene	8100, 8270, 8310	0.03 mg/kg
Acenaphthylene	" "	" "

<b>Table 6.1. Possible Chemicals to be Analyzed from Sediment Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit</b>
Anthracene	8100, 8270, 8310	0.03 mg/kg
Benzo(a)Anthracene	" "	" "
Benzo(a,e)Pyrene	" "	" "
Benzo(g,h,i)Perylene	" "	" "
Benzo(k)Fluoranthene	" "	" "
Benzo(b)Fluoranthene	" "	" "
Chrysene	" "	" "
Dibenzo(a,h)Anthracene	" "	" "
Fluoranthene	" "	" "
Fluorene	" "	" "
Indeno(1,2,3,4,-c,-d) Pyrene	" "	" "
Methylnaphthalene	" "	" "
Naphthalene	" "	" "
Phenanthrene	" "	" "
Pyrene	" "	" "
<b>Organotin Compounds</b>		
Monobutyltin	Stephensen & Smith, 1988 or Uhler & Durrel, 1989	0.01 mg/kg
Dibutyltin	" "	" "
Tributyltin	" "	" "
<b>Phenols and Substituted Phenols</b>		
Phenol	8040,8270	0.01-1.5 mg/kg
2,4-dimethylphenol	" "	" "
2,4,6-trichlorophenol	" "	" "

<b>Table 6.1. Possible Chemicals to be Analyzed from Sediment Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit</b>
Para-chloro-meta-cresol	8040, 8270	0.01-1.5 mg/kg
2-chlorophenol	" "	" "
2,4, dichlorophenol	" "	" "
2-nitrophenol	" "	" "
4-nitrophenol	" "	" "
2,4-dinitrophenol	" "	" "
4,6-dinitro-o-cresol	" "	" "
Pentachlorophenol	" "	" "
<b>Phthalate Esters</b>	" "	" "
Bis(2-Ethylhexyl)Phthalate	8060	0.1 mg/kg
Butylbenzyl Phthalate	" "	" "
Diethyl Phthalate	" "	" "
Dimethyl Phthalate	" "	" "
Di-n-Butyl Phthalate	" "	" "
<b>Polychlorinated Biphenyls (PCB's)</b>		
PCB-1016	Tetra Tech, 1986a and NOAA 1989;8080	0.01 mg/kg
PCB-1221	" "	" "
PCB-1232	" "	" "
PCB-1242	" "	" "
PCB-1248	" "	" "
PCB-1254	" "	" "
PCB-1260	" "	" "

<sup>1</sup> EPA method unless otherwise noted

## **7.0 BIOASSAY AND BIOACCUMULATION TESTING OF DREDGED MATERIAL**

Bioassay tests (in Tier III) must be conducted on all proposed dredging, reference and control site samples, if required, according to the protocol outlined in the 1991 Green Book. Strict adherence to the 1991 Green Book bioassay procedures is required. Any deviations from the procedures must be agreed to by the CE District and EPA Region IV prior to testing. Bioassay and bioaccumulation testing will be conducted according to test conditions listed in Appendix D. In the event that laboratory test conditions are not established yet, documentation will be provided to the CE District according to Appendix E. In this case, laboratory conditions, for proposed testing must be approved, by the CE District and EPA, prior to testing.

### **7.1 Water - Column Acute Toxicity Tests**

Table 7.1 lists recommended tests species for conducting the Water - Column Acute Toxicity Bioassay. Test species must include at least 2 test species living in the water column.

### **7.2 Whole - Sediment Bioassay and Bioaccumulation Tests**

The whole - sediment tests, both bioassay and bioaccumulation, must be conducted with at least two different, appropriately sensitive, marine benthic species covering the three species characteristics (i.e., the test organisms include species representative of a filter-feeder, deposit-feeder and at least one species which burrows) described in 40 CFR 227.27 (d) (See Tables 7.2 & 7.3). The use of one amphipod test species is required when conducting whole - sediment bioassays. Bioaccumulation tests must be conducted on all reference and proposed dredged material samples according to protocols described in the 1991 Green Book. The length of time for bioaccumulation tests is 10 days if only heavy metal contamination is suspected or 28 days if organic chemical contamination is suspected. If both heavy metal and organic metal contamination is suspected then tests will be conducted for 28 days. The use of one polychaete species is required when conducting bioaccumulation tests.

### **7.3 Possible Chemicals to be Analyzed from Tissue Samples**

Tissues from the test organisms used in the bioaccumulation tests may be analyzed as deemed necessary for the chemicals listed in Table 7.4. The chemicals to be analyzed for in the test organisms tissues will be based on the Tier I evaluation and the results from bulk sediment chemistry analyses conducted on proposed dredged material.



**Table 7.1 Recommended Test Species for Water - Column Toxicity Testing of Dredged Material.**

**Oyster Larvae**

*Crassostrea virginica*

**Sea Urchin Larvae**

*Arbacia punctulata*

**Lytechinus pictus**

**Crustaceans**

*Mysidopsis bahia*

*Mysidopsis bigelowi*

*Mysidopsis almyra*

**Fishes**

**Silversides**

*Menidia menidia*

*Menidia beryllina*

*Menidia peninsulae*

**Sheepshead Minnow**

*Cyprinodon variegatus*

**Table 7.2. Recommended Test Species for Whole - Sediment Bioassay Testing of Dredged Material.**

**Infaunal Amphipods**

*Rhepoxinius abronius* D, F

*Eohaustorius estuarius* D, F

*Ampelisca abdita* D, F

**Mysid Shrimp**

*Mysidopsis almyra* D, F

*Mysidopsis bahia* D, F

*Mysidopsis bigelowi* D, F

**Commercial Shrimp**

*Penaeus aztecus* D, B

*Penaeus duorarum* D, B

*Penaeus setiferus* D, B

**Burrowing Polychaetes**

*Neanthes succinea* D, B

*Neanthes virens* D, B

*Arenicola cristata* D, B

**Juvenile Bivalves**

*Macoma* spp. F, B

*Rangia cuneata* F, B

*Geukensia demissa* F, B

B - Burrower, D - Deposit Feeder, F - Filter Feeder

**Table 7.3. Recommended Test Species for Determining Potential Bioaccumulation of Dredged Material.**

**Shrimp**

*Penaeus aztecus* D, B

*Penaeus duorarum* D, B

*Penaeus setiferus* D, B

**Burrowing Polychaetes**

*Neanthes succinea* D, B

*Neanthes virens* D, B

*Arenicola cristata* D, B

**Bivalves**

*Mercenaria mercenaria* F, B

*Rangia cuneata* F, B

*Geukensia demissa* F, B

*Macoma* spp. F, B

B - Burrower, D - Deposit Feeder, F - Filter Feeder

<b>Table 7.4. Possible Chemicals to be Analyzed from Tissue Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit<sup>2</sup></b>
<b>Tissue Characteristics</b>		
Total Lipids	Lee et al. 1989	0.1%
Total Water Content	EPA 1986a, 1987a	0.1%
<b>Metals</b>		
Antimony	7040, 7041	0.50 mg/kg
Arsenic	7060, 7061	0.10 mg/kg
Beryllium	7090, 7091	" "
Cadmium	7130, 7131	" "
Chromium	7190, 7191	" "
Copper	7210, 7211	" "
Lead	7420, 7421	" "
Mercury	7471	0.05 mg/kg
Nickel	7520, 7521	0.10 mg/kg
Selenium	7740, 7741	0.20 mg/kg
Silver	7760, 7761	0.10 mg/kg
Thallium	7840, 7841	" "
Zinc	7950, 7951	" "
<b>Nonmetals</b>		
Cyanide	9010, 9012	1.0 mg/kg
2,3,7,8- TCDD, TCDF & Congeners	8290	1 ng/kg
Total Sulfides	9030, Plumb 1981	0.20 mg/kg
<b>Pesticides</b>		
Aldrin	8080	0.03 mg/kg
Chlordane & Derivatives	" "	" "

<b>Table 7.4. Possible Chemicals to be Analyzed from Tissue Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit<sup>2</sup></b>
Diieldrin	8080	0.03 mg/kg
DDT & Derivatives	" "	" "
Endosulfan & Derivatives	" "	0.02 mg/kg
Endrin & Derivatives	" "	0.01 mg/kg
Heptachlor & Derivatives	" "	0.02 mg/kg
Hexachlorocyclohexane & Derivatives	" "	0.01 mg/kg
Methoxychlor	" "	0.02 mg/kg
Toxaphene	" "	0.01 mg/kg
<b>Organic Compounds</b>		
Polynuclear Aromatic Hydrocarbons (PAH's)		
Acenaphthene	8100, 8270, 8310	0.03 mg/kg
Acenaphthylene	" "	" "
Anthracene	" "	" "
Benzo(a)Anthracene	" "	" "
Benzo(a,e)Pyrene	" "	" "
Benzo(g,h,i)Perylene	" "	" "
Benzo(k)Fluoranthene	" "	" "
Benzo(b)Fluoranthene	" "	" "
Chrysene	" "	" "
Dibenzo(a,h)Anthracene	" "	" "
Fluoranthene	" "	" "
Fluorene	" "	" "
Indeno(1,2,3,4,-c,-d) Pyrene	" "	" "

<b>Table 7.4. Possible Chemicals to be Analyzed from Tissue Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit<sup>2</sup></b>
Methylnaphthalene	8100, 8270, 8310	0.03 mg/kg
Naphthalene	" "	" "
Phenanthrene	" "	" "
Pyrene	" "	" "
<b>Organotin Compounds</b>		
Monobutyltin	Stephensen & Smith, 1988 or Uhler & Durrel, 1989	0.01 mg/kg
Dibutyltin	" "	" "
Tributyltin	" "	" "
<b>Phenols and substituted Phenols</b>		
Phenol	8040, 8270	0.1-1.5 mg/kg
2,4-dimethylphenol	" "	
2,4,6-trichlorophenol	" "	" "
Para-chloro-meta-cresol	" "	" "
2-chlorophenol	" "	" "
2,4,-dichlorophenol	" "	" "
2-nitrophenol	" "	" "
4-nitrophenol	" "	" "
2,4-dinitrophenol	" "	" "
4,6-dinitro-o-cresol	" "	" "
Pentachlorophenol	" "	" "
<b>Phthalate Esters</b>		
Bis(2-ethylhexyl)phthalate	8060	0.1 mg/kg

<b>Table 7.4. Possible Chemicals to be Analyzed from Tissue Samples.</b>		
<b>Chemical</b>	<b>Test Method<sup>1</sup></b>	<b>Method Detection Limit<sup>2</sup></b>
Butyl benzyl phthalate	8060	0.1 mg/kg
Di-n-butyl phthalate	" "	" "
Di-n-octyl phthalate	" "	" "
Diethyl phthalate	" "	" "
Dimethyl phthalate	" "	" "
<b>Polychlorinated Biphenyls (PCB's)</b>		
PCB-1016	8080, NOAA 1989	0.01 mg/kg
PCB-1221	" "	" "
PCB-1232	" "	" "
PCB-1242	" "	" "
PCB-1248	" "	" "
PCB-1254	" "	" "
PCB-1260	" "	" "

<sup>1</sup> EPA unless otherwise noted

<sup>2</sup> Method Detection Limits are based on a sample size of 30 grams (wet weight)

## **8.0 STATISTICAL ANALYSES**

Following sampling and testing, statistical analyses of the results may be necessary to determine the suitability of the proposed dredged material for ocean disposal. In the case that non-detects (i.e., values below the method detection limits) occur, these values should be set at one half the appropriate detection method detection limit prior to statistical analysis. Coordination with the CE District, while analyzing the test data, is recommended and complete documentation of statistical analyses should be supplied to the CE District. The approaches to the statistical analysis of dredged material test results are currently undergoing reevaluation. Upon completion, these revisions will be included in subsequent RIM editions. Permit applicants are encouraged to consult appropriate statistical references as needed.

### **8.1 Determination of the Appropriate Statistical Test: the Assumptions**

The first step in determining which statistical test to employ is to assess whether the data will meet the assumptions of the selected parametric or nonparametric test. If the assumptions can be satisfied, parametric tests are preferred since these tests are a more powerful form of analysis.

#### **8.1.1 The Normality Assumption**

The normality and homogeneity of variance assumptions (see below) can be evaluated at the same time using basic descriptive statistics. Normality is usually examined using simple plots of the data. In the event that the data are not normally distributed, appropriate transformations of the data should be considered (see section 8.2). If the transformed data still do not satisfy this assumption then an appropriate nonparametric test should be considered.

#### **8.1.2 The Homogeneity of Variance Assumption**

The homogeneity of variance assumption must be tested when using Analysis of Variance (ANOVA). This assumption can be tested using either Cochran Q, Bartlett's test for homogeneity of variances, or Hartley's  $F_{\max}$ -test. These tests are available in most of the commonly used computer assisted packages. As with the normality assumption, data transformation may be required to satisfy this assumption.

### **8.2 Data Transformations**

If the assumptions of the preferred statistical test can not be satisfied the data should either be transformed in such a manner that the assumptions can be satisfied. The most commonly used transformations are listed in this section. The use of any other transformation must be approved by the EPA and CE District prior to use. Often a single transformation can simultaneously satisfy several assumptions. Following transformation, the data should be re-examined to determine if the test assumptions are met. In the event that test assumptions are still not met following transformation, data should be analyzed using a distribution free statistical (nonparametric) test



should be used.

### **8.2.1 Log Transformation**

The most commonly used transformation is the logarithmic transformation. This transformation will make the variance independent of the mean thus providing an approximate normal transformation.

### **8.2.2 Arcsin Transformation**

This transformation is required if the data are percentages or proportions.

### **8.2.3 Square Root Transformation**

If the data are counts, the square root transformation is preferred. Transformations of these types of data to square roots will also make the variances independent of the means. When the data are counts which include zeros then a constant (i.e., 0.5 or 1.0) should be added to each data point prior to finding the square root.

## **8.3 Analysis of Water - Column Bioassay Test Results**

The two sample t-test is the preferred statistical test to be used to detect survival differences between the control water and dilution water treatments following water column bioassay testing (EPA and CE, 1991). Use of the t-test depends on the assumption that the two treatments have equal variances. In the event of unequal variances analyses, the t-test should be conducted as described on page 13-5 of the Green Book. If concern warrants further analysis, a Mann - Whitney U test or a Wilcoxon Rank Sum test should be used.

## **8.4 Analysis of Whole - Sediment Bioassay, Bioaccumulation, and Physical Parameter Test Results**

The t-test is also used to analyze data collected during tier III benthic bioassay, and bioaccumulation testing, at the reference and ODMDS sites. Again the use of the t-test depends on the assumption that the two treatments have equal variances. In the event of unequal variances analyses, the t-test should be conducted as described on page 13-5 of the Green Book. If concern warrants further analysis, a Mann - Whitney U test or a Wilcoxon Rank Sum test should be used.

## 9.0 QUALITY CONTROL AND ASSURANCE

This section provides guidance to ensure the quality of the data collected during the laboratory phase of studies related to the ocean disposal of dredged materials. This section is modified from EPA draft guidelines for laboratory quality assurance (QA) and quality control (QC) (EPA 1992).

The importance of a QA program to dredging studies is to ensure that collected data, required to make permitting decisions, is of known and documented quality. QA activities also ensure that quality control (QC) procedures have been implemented and documented. QA programs set standards for personnel qualifications, facilities, equipment, services, data generation, record keeping, and data-quality assessments. The function of a government QA program is to ensure that contracted laboratories comply with procedures in the 1991 Green Book (see Chapter 14 in EPA and CE 1991, and EPA 1987c) and this RIM. The QA oversight is the responsibility of the QA coordinator in the pertinent CE district. QA oversight is carried out in three ways: (1) preaward inspections; (2) interlaboratory comparisons; and (3) routine inspections during the conduct of the study.

QC is also an integral part of any dredging study. QC plans include: measurements of data quality using blanks, spikes, and control test group results which are compared with test results from dredging studies. Chemical QC specifications include the acceptable ranges for instrument calibration, analyte recovery, data accuracy, and precision. Certain samples may be required to be submitted on a routine basis to government laboratories for analysis. Biological QC involves periodic reference toxicant testing with stock organisms used in dredged material tests.

The following sections detail the Quality Assurance/Quality Control (QA/QC) procedures recommended to ensure that only the highest-quality data are used in determining the suitability of dredged material for disposal in the ocean. These QA/QC guidelines draw on components from several programs, including, but not limited to, the U.S. EPA Environmental Monitoring and Assessment Program (EMAP), the National Status and Trends Program and the Puget Sound Estuary Program. These guidelines reflect a "performance based" approach.

The first phase of a "performance based" program is an initial demonstration of capability or performance evaluation. Prior to sample analysis, the laboratory must demonstrate proficiency in several ways including: (1) providing written protocols for the analytical methods to be employed for sample analysis; (2) calculating method detection limits for each analyte; (3) establishing an initial calibration curve for all analytes; and generally; (4) demonstrating acceptable performance on known or blind accuracy-based material. Following a successful first phase, the laboratory must also demonstrate its continued capability in several ways including: (1) participation in refereed intercomparison exercises; (2) repeated analysis of certified reference materials; (3) calibration checks; and (4) analysis of laboratory reagent blanks and fortified samples. These steps are detailed in the following sections and are summarized in Table 9.2. The sections are arranged to mirror the elements in Table 9.2 allowing users to more easily cross reference the specific details with the general topics in the table.

## **9.1 General QA/QC Requirements**

The guidance provided in the following sections is based largely on the protocols developed for the Puget Sound Estuary Program (EPA 1989) and the EMAP Program; with method detection limits reaching the low parts-per-billion for both sediment and tissue analyses unless otherwise noted. The QA/QC requirements provide a common foundation for each laboratory's protocols, enabling an assessment of the comparability of results generated by different laboratories and analytical procedures. It should be noted that the specified QA/QC requirements in this plan represent the minimum requirements for any given analytical method. Additional requirements which are method specific should also be followed, as long as the minimum requirements presented in this document have been met.

The results for the various QA/QC samples must be reviewed by laboratory personnel immediately following the analysis of each sample batch. These results should then be used to determine if warning and control limits have been exceeded and corrective actions must be taken, before processing a subsequent sample batch (Table 9.2). Warning limits are numerical criteria that serve as flags to data reviewers and users. When a warning limit is exceeded, the laboratory is not obligated to halt analyses, but the reported data may be qualified during subsequent QA/QC review. Control limits are numerical data criteria that, when exceeded, require specific corrective action by the laboratory before subsequent analyses proceed. Warning and control limits and the recommended frequency of analysis for each QA/QC element or sample type are summarized in Table 9.2. Descriptions of the use, frequency of analysis, type of information obtained, and corrective actions for each of these QA/QC sample types or elements are provided in the following sections.

## **9.2 Initial Demonstration of Capability**

A laboratory's initial demonstration of capability should include: written protocols for sample analysis; the calculation of method detection limits for each analyte; the establishment of an initial calibration curve for each analyte; and if possible, documentation of acceptable performance on a "performance evaluation" sample. These components are described in the following paragraphs.

### **9.2.1 Initial Calibration**

Equipment must be calibrated before any samples are analyzed, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended control limit criteria (Table 9.2). All calibration standards should be traceable to a recognized organization for the preparation of QA/QC materials (e.g., National Institute of Standards and Technology (NIST), U.S. Environmental Protection Agency, etc.). Calibration curves must be established for each element and batch analysis from a calibration blank and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. The calibration curve must be established prior to the analysis of samples. Only data within the demonstrated working calibration range may be reported by the laboratory; samples outside this range should be diluted or concentrated, as appropriate, and reanalyzed.

<b>Table 9.2 Key elements for quality control of chemical analyses.</b>			
<b>Element or Sample Type</b>	<b>Warning Limit Criteria</b>	<b>Control Limit Criteria</b>	<b>Frequency</b>
<b>Initial Demonstration of Capability (Prior to Sample Analyses):</b>			
-initial calibration	NA	NA	initially then prior to analyzing each sample batch
-calculation of method detection limits	must be equal to or less than target values (see Table 6.1)		at least once each project
- blind analysis of accuracy based material	NA	NA	initial
<b>On-going Demonstration of Capability:</b>			
-blind analysis of laboratory intercomparison exercise samples	NA	NA	regular intervals throughout project
-continuing calibration checks using calibration standard solutions	NA	should be within $\pm 15\%$ of initial calibration on average for all analytes, not to exceed $\pm 25\%$ for any one analyte	at a minimum, middle and end of each batch
-analysis of Certified Reference Material (CRM) or Laboratory Control Material (LCM):			one with each batch of samples
Precision <sup>1</sup>	NA	value obtained for each analyte should be within 3 standard deviations of control chart limits	value plotted on control chart after each analysis of CRM

<b>Table 9.2 Key elements for quality control of chemical analyses.</b>			
<b>Element or Sample Type</b>	<b>Warning Limit Criteria</b>	<b>Control Limit Criteria</b>	<b>Frequency</b>
<b>Recovery Accuracy<sup>2</sup></b>			
- PAH' s	lab values should be within $\pm 25\%$ of true value on average for all analytes; not to exceed $\pm 30\%$ of true value for more than 30% of individual analytes	lab values should be within $\pm 30\%$ of true value on average for all analytes; not to exceed $\pm 35\%$ of true value for more than 30% of individual analytes	
- PCB's/pesticides	same as above	same as above	
-inorganic elements	lab should be within $\pm 15\%$ of true value for each analyte	lab should be within $\pm 20\%$ of true value for each analyte	
-laboratory reagent blank	analysts should use best professional judgement if analytes are detected at $< 3$ times the MDL	no analyte should be detected at $> 3$ times the MDL	one with each batch of samples
-laboratory fortified sample matrix (matrix spike) <sup>4</sup>	NA	recovery should be within the range 50 to 120% for at least 80% of the analytes	at least 5% of total number of samples
-laboratory duplicate or sample matrix duplicate (matrix spike duplicate)	NA	RPD <sup>3</sup> must be $\leq 30$ for each analyte	same as matrix spike
-internal standards (surrogates)	NA	recovery must be within the range 30 to 150%	each sample
-internal injection standards	lab develops its own		each sample

<sup>1</sup> The use of control charts to monitor precision for each analyte of interest should follow generally accepted practices (e.g., Taylor 1987). Upper and lower control limits, based on three standard

deviations (3 std) of the means should be updated at regular intervals.

<sup>2</sup> "True" values in CRM's may be either "certified" or "non-certified" (it is recognized that absolute accuracy can only be assessed using-certified values, hence the term relative accuracy). Relative accuracy is computed by comparing the laboratory's value for each analyte against either end of the range of values (i.e., 95 % confidence limits) reported by the certifying agency. The laboratory's value must be within 35% of either the upper or low 95 % confidence interval value. Accuracy control limit criteria only apply for analytes having CRM concentration 210 times the laboratory's MDL.

<sup>3</sup> RPD = Relative percent difference between matrix spike and matrix spike duplicate results (see section 9.3.6 Laboratory Duplicates for equation).

<sup>4</sup> Samples to be spiked should be chosen at random; matrix spike solutions should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be at least 10 times the calculated MDL.

### 9.2.2 Calculation of Method Detection Limits

Analytical chemists have coined a variety of terms to define "limits" of detectability; definitions for some of the more commonly used terms are provided in Keith *et al.* (1983) and in Keith (1991). In this document, the Method Detection Limit (MDL) will be used to define the analytical limit of detectability. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: "the MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." Confidence in the apparent analyte concentration increases as the analyte signal increases above the MDL.

Each analytical laboratory should calculate and report an MDL for each analyte of interest in each matrix of interest (sediment or tissue) prior to the analysis of samples. Each laboratory should follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate MDL's for each analytical method employed. The matrix and the amount of sample used in calculating the MDL should match as closely as possible - the matrix of the actual samples and the amount of sample typically used. In order to ensure comparability of results among different laboratories, MDL target values have been recommended (Table 6.1). The initial MDL's reported by each laboratory should be equal to or less than these specified target values before the analysis of samples may proceed. It is recognized that the initial MDL is a statistically-derived, empirical value that may vary in actual samples as a function of the sample matrix, volume, percent moisture, etc. Each laboratory must periodically (i.e., at least once each year) re-evaluate its MDL's for the analytical methods used and the sample matrices typically encountered.

### **9.2.3 Blind Analysis of Accuracy-Based Material**

Whenever possible, a representative sample matrix which is uncompromised, homogeneous and contains the analytes of interest should be analyzed blind by each laboratory. The purpose of analyzing the sample(s) "blind" (where the laboratory does not know the concentrations of the analytes) is to assess the accuracy of the laboratory's performance prior to the analysis of actual field samples. Typically, an SRM or CRM is used for this "blind" sample. A laboratory's performance is then determined by comparing the analytical results to the certified concentrations. Acceptable performance is generally indicated by concentrations that are  $\pm 30\%$  for organic analytes and  $\pm 20\%$  for inorganic analytes of the known concentration of the analytes in the sample. These criteria are recommended only for analyte concentrations that are equal to or greater than 10 times the MDL as established by the laboratory. Failure of the laboratory to meet these criteria should result in reanalysis of the sample until acceptable performance is obtained.

## **9.3 On-going Demonstration of Capability**

In order to ensure that the laboratory is consistently producing comparable high-quality data during a project, the following QC elements are suggested: participation in intercomparison exercises, continuing calibration checks, analysis of reference materials, reagent blanks, matrix spikes and duplicate samples, monitoring internal and injection internal standard performance. Criteria for warning and control limits are presented in Table 9.2, and discussion related to each element is presented in the following sections.

### **9.3.1 Laboratory Participation in Intercomparison Exercises**

The laboratory intercomparison exercises previously referred to are sponsored by the NOAA National Status and Trends Program (NS&T) to evaluate both the individual and collective performance of its participating analytical laboratories. However, it may be difficult for other laboratories to participate in these exercises. Therefore this section may not be applicable. It is highly recommended that each laboratory include some type of intercomparison exercise in their QA/QC program. Typically, three or four different NS&T exercises are conducted over the course of a year; each exercise involves the blind analysis of different representative matrices (e.g., standard solutions, sediment or tissue samples) distributed to all laboratories in common by either NIST or National Research Council of Canada (under contract to NOAA). Following the initial demonstration of capability, each laboratory is required to participate in these on-going intercomparison exercises as a continuing check on performance and intercomparability. Laboratories which fail to achieve acceptable performance in any intercomparison exercise must provide an explanation and may be required to undertake corrective actions.

### **9.3.2 Continuing Calibration Checks**

The initial instrument calibration is checked through the analysis of a calibration standard. If possible, the calibration standard solution used for the calibration check should be obtained from a

different source than the initial calibration standards, so that it can provide an independent check both on the calibration and the accuracy of the standard solutions. Analysis of the calibration standard should occur at the beginning of a sample set (i.e., batch), at least once every 10 samples, and after the last sample in the batch.

If the control limit for analysis of the calibration standard is not met (Table 9.2), the initial calibration will have to be repeated. If possible, the samples analyzed before the calibration check that failed the control limit criteria should be reanalyzed following the re-calibration. The laboratory should begin by reanalyzing the last sample analyzed before the calibration standard which failed. If the relative percent difference (RPD) between the results of this reanalysis and the original analysis exceeds 30 percent, the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples should progress in reverse order until it is determined that there is less than 30 RPD between initial and reanalysis results. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) is considered suspect. It is also possible that the majority of analytes will meet the calibration criteria, while only a few may not. In this case, the best professional judgement of the analyst may be required to assess the acceptability of the calibration. In this case, the laboratory should include a written narrative with the data describing the situation.

### **9.3.3 Routine Analysis of Reference Materials**

Reference Materials (SRM's or CRM's) generally are considered the most useful QC samples for assessing the accuracy of a given analysis (i.e., the closeness of a measurement to the "true" value). Because Certified Reference Materials have "certified" concentrations of the analytes of interest, as determined through replicate analyses using two independent measurement techniques, these materials can be used to assess accuracy. Thus, routine analysis of reference materials thus represents a particularly vital aspect of the "performance-based" QA philosophy.

A Laboratory Control Material (LCM) is similar to a Certified Reference Material in that it is a homogeneous matrix which closely matches the samples being analyzed. A "true" LCM is one which is prepared (i.e., collected, homogenized and stored in a stable condition) strictly for use in-house by a single laboratory. Alternately, the material may be prepared by a central laboratory and distributed to others (so-called regional or program control materials). Unlike CRM's, concentrations of the analytes of interest in LCM's are not certified but are based upon a statistically-valid number of replicate analyses by one or several laboratories. In practice, this material can be used to assess the precision (i.e., consistency) of a single laboratory, as well as to determine the degree of comparability among different laboratories. If available, LCM's may be preferred for routine (i.e., day-to-day) analysis because CRM's are relatively expensive. However, CRM's still must be analyzed at regular intervals (e.g., monthly or quarterly) to provide a check on accuracy. One SRM, CRM or LCM should be analyzed along with each batch of 20 or fewer samples (Table 9.2). The SRM, CRM or LCM concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on accuracy for each batch of samples before proceeding with a subsequent batch. If values are outside the control



limits (Table 9.2), the data for the entire batch of samples is considered suspect. Calculations and instruments should be checked; the control material may have to be reanalyzed (i.e., reinjected) to confirm the results. If the control limits are still exceeded in the repeat analysis, the laboratory is required to determine the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing. The results of the CRM or LCM analysis should not be used by the laboratory to "correct" the data for a given sample batch.

Results of control material analyses also should be recorded on control charts to monitor laboratory precision from batch to batch. This is particularly important in situations where certified concentrations are not available for all the analytes of interest in a particular SRM or CRM. In such instances, each Laboratory should be able to demonstrate an acceptable level of batch-to-batch consistency for a given reference material, in accordance with commonly employed control charting techniques (i.e., wildly fluctuating results are not acceptable).

The "absolute" accuracy of an analytical method can be assessed using CRM's only when certified values are provided for the analytes of interest. However, the concentrations of many analytes of interest are provided only as non-certified values in some of the more commonly used CRM's. Therefore, control limit criteria are based on "relative accuracy", which is evaluated for each analysis of the CRM or LCM by comparison of a given Laboratory's values relative to the "true" or "accepted" values in the LCM or CRM. In the case of CRM's, this includes both certified and noncertified values and encompasses the 95 % confidence interval for each value as described in Table 9.2.

Accuracy control limit criteria have been established both for individual compounds and combined groups of compounds (Table 9.2). There are two combined groups of compounds for the purpose of evaluating relative accuracy for organic analyses: PAH's and PCB's/pesticides. The laboratory's value should be within  $\pm 30\%$  of the true value on average for each combined group of organic compounds, and the laboratory's value should be within  $\pm 35\%$  of either the upper or lower 95 % confidence limit for at least 70% of the compounds in each group. For inorganic analyses, the laboratory's value should be within  $\pm 20\%$  of either the upper or lower 95 % confidence limit for each analyte of interest in the CRM. Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes having CRM true values which are  $\geq 10$  times the MDL established by the laboratory.

#### **9.3.4 Laboratory Reagent Blank**

Laboratory reagent blanks (commonly called method blanks) are used to assess contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one reagent blank should be run in every sample batch (minimum frequency of one per 20 samples). Warning and control limits for blanks (Table 9.2) are based on the laboratory's method detection limits as documented prior to the analysis of samples. A reagent blank concentration between the MDL and three times the MDL should series as a warning limit

requiring further investigation based on the best professional judgement of the analyst(s). A reagent blank concentration equal to or greater than three times the MDL requires definitive corrective action to identify and eliminate the source(s) of contamination.

### **9.3.5 Laboratory Fortified Sample Matrix**

A laboratory fortified sample matrix (commonly called a matrix spike) should be used to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest. A minimum of 5 % of the total number of samples submitted to the laboratory in a given project should be selected at random for analysis as laboratory fortified samples. The compounds used to fortify samples should include all the analyte of interest. These compounds should be added at 5 to 10 times their MDL's as previously calculated by the laboratory.

The recovery data for each fortified compound, which should be reported along with the rest of the data for each sample, ultimately will provide additional information on the performance of the methods. If the percent recovery for any analyte is less than the recommended warning limit of 50 percent, the chromatograms and/or raw data quantitation reports should be reviewed. Corrective actions taken and verification of acceptable instrument response should be included. The laboratory should document the recoveries in a control chart as a long-term assessment of method, and laboratory performance.

### **9.3.6 Laboratory Duplicates**

One sample per batch should be split in the laboratory and analyzed in duplicate to provide an estimate of analytical precision. Duplicate analyses also are useful in assessing potential sample heterogeneity and matrix effects. An alternative to a sample duplicate is a matrix spike duplicate. If results fall outside the control limit (Table 9.2), calculations and instrument should be checked. A replicate analysis may be required to confirm the results. If results continue to exceed the control limit, subsequent collective action is at the discretion of the program manager or QA officer, because matrix effects or incomplete homogenization (either in the field or laboratory) may be contributing factors. The relative percent difference (RPD) between the analytical results for the duplicate samples (or matrix spike and matrix spike duplicate) should be less than 30 for each analyte of interest (Table 9.2). The RPD is calculated as follows:

$$RPD = \frac{(C_1 - C_2) \times 100\%}{(C_1 + C_2)/2}$$

where:  $C_1$  is the larger of the duplicate concentrations for a given analyte  
 $C_2$  is the smaller of the duplicate concentrations for a given analyte.

If results for any analytes do not meet the recommended  $RPD \pm 30\%$  control limit criteria, calculations and instrumentation should be checked. It may be necessary to repeat the analysis to confirm the results. Results which repeatedly fail to meet the control limit criteria may indicate

poor laboratory precision. If this is the case, the laboratory should halt analysis of samples and eliminate the source of the imprecision before proceeding with sample analysis.

### 9.3.7 Internal Standards

Internal standards (commonly referred to as surrogate spikes or surrogate analyses) are compounds chosen to simulate the analytes of interest in organic analyses. Ideally, the internal standard(s) is an isotopically-labeled analog of an analyte, although this type of standard is suitable for GC/MS analysis only. Alternatively, a compound similar to the analytes to be quantitated should be used as the internal standard(s). PCB congeners 198 and 103 have been successfully utilized as internal standards for PCB quantitation due to their relative retention indices and extremely low concentrations in environmental samples. Similarly, gammachlordene has been used to quantitate the more polar pesticides. Wherever possible, the use of multiple internal standards which elute at dramatically different times (e.g. congeners 103 and 198 which elute toward the beginning and end of a GC run, respectively) is highly recommended. The use of multiple internal standards may provide better quantitations by minimizing response differences caused by such factors as automatic injections.

The internal standard represents a reference against which the signal from the analytes of interest is compared directly for the purpose of quantification. Internal standards must be added to each sample, including QA/QC samples, prior to extraction. The internal standard recovery therefore should be carefully monitored; each laboratory should report the absolute amounts and the percent recovery of the internal standards along with the target analyte for each sample. Using this approach, the analytes of interest are assumed to behave identically to the appropriate internal standard(s). The internal standard is assumed to be fully recovered in an internal standard type calibration. Even if this assumption is not valid, as based on the use of an external standard, as described below, it is still assumed that the analytes of interest behave (i.e., are not fully recovered) in the same manner as the internal standard(s). Therefore, the ratio of the concentration of the analytes to the concentration of the internal standard is constant. Recovery of the internal standard(s) is determined through the use of an external or internal injection standard as described below. Acceptable internal standard recoveries are listed in Table 9.2. These limits are asymmetrical because low recoveries are provided for as described above, while recoveries greater than 100% for the internal standard(s) may indicate an interference with the internal standard(s) which could affect data quality.

### 9.3.8 Internal Injection Standards

For gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS) analysis, internal injection standards are added to each sample just prior to injection. Internal injection standards are used to monitor the actual recovery of the internal standards. The analyst(s) should monitor internal injection standard retention times and response to determine if instrument maintenance or repair is needed. Instrument problems that may have affected the data or resulted in the analysis of the sample should be documented properly in logbooks and/or

internal data reports and used by the laboratory personnel to take appropriate corrective action.

## 10.0 REFERENCES

In the event that more detailed methodological investigations are needed the following references should be consulted by MPRSA Section 103 permit applicants:

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- (EPA) U.S. Environmental Protection Agency. 1987b. Evaluation of surveying positioning methods for nearshore marine and estuarine waters. EPA 430/9-87-003.
- (EPA) U.S. Environmental Protection Agency. 1987c. Quality assurance/quality control (QA/QC) for 301(h) monitoring programs: Guidance on field and laboratory methods. EPA 430/9-86-004.
- (EPA) U.S. Environmental Protection Agency. 1987d. Technical Support Document for ODES statistical power analysis. 430/9-87-005.
- (EPA) U.S. Environmental Protection Agency. 1987e. Quality Criteria for water - 1986 (the Gold Book). Office of water regulations and standards, May 1, 1987 (including updates 1 & 2) EPA 440/5-86-001.
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## Appendix A

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## **Appendix B**

### **MPRSA Ocean Disposal Evaluation Documentation**

The following information is required for completion of the MPRSA Section 103 evaluation. Information should not be repeated but referenced where material is needed for more than one part of the evaluation documentation.

#### **1. Dredging and Disposal Project Information**

- a. a map showing dredging locations/boundaries
- b. core boring logs keyed to the map (if available)
- c. volume of material to be dredged
- d. percentages of fine, medium and coarse grained material by dredging unit
- e. bathymetric information for the channel to be dredged (if available)
- f. design depth and width
- g. expected method(s) of dredging, transport and disposal of material
- h. expected start, duration and end of dredging, transport and disposal of material
- i. location of placement of dredged material at the ODMDS
- j. compliance with ODMDS site designation conditions (if available)

#### **2. Exclusionary Criteria and Need for Testing Documentation**

- (i) rationale for meeting exclusionary criteria
- (ii) locations (keyed to a map), quantities and types of pollutants discharged upstream of the dredging area (if available)
- (iii) grain sizes of the dredged material (from 1d above)
- (iv) results and dates of previous testing (if available)
- (v) dates of previous dredging

#### **3. Water - Column Determinations (Tiers II-IV)**

- a. Limiting Permissible Concentration Compliance Documentation
  - (i) results of the ADDAMS model
  - (ii) comparison with water quality criteria
- b. Water - Column Toxicity Evaluation
- c. Water - Column Testing Report

#### **4. Benthic Determinations (Tiers II-IV)**

- a. Benthic Toxicity Evaluation
- b. Benthic Bioaccumulation Evaluation
  - (i) Theoretical Bioaccumulation Potential
  - (ii) Benthic Bioavailability Evaluation
- c. Sediment Testing Report (see Appendix C of this document for detailed format)

5. MPRSA Section 103 Ocean Disposal Criteria Compliance Evaluation
  - a. Compliance with Part 227 Subpart B - Environmental Impact
    - (i) 227.4 criteria
    - (ii) 227.5 prohibited materials
    - (iii) 227.6 prohibited constituents
    - (iv) 227.9 limitations on quantities
    - (v) 227.10 hazards
    - (vi) 227.13 dredged material
  - b. Compliance with Part 227 subpart C - Need for Ocean Dumping
    - (i) all sections
  - c. Compliance with Part 227 subpart D - Impact of the Proposed Dumping on Esthetic, Recreational and Economic Values
    - (i) all sections
  - d. Compliance with Part 227 subpart E - Impact of the Proposed Dumping on other Uses of the Ocean
    - (i) all sections
6. Requirements (Management Options) to meet Ocean Disposal Criteria (if applicable).
7. Requirements of Site Designation Conditions (if applicable).
8. MPRSA Section 103 Conditions.

## **Appendix C**

### **Sediment Testing Report Format**

The sediment physical, chemical, bioassay and bioaccumulation test results should be reported in the following format:

#### **A. Abstract**

#### **B. Introduction**

1. Project description enclosing: large scale map showing the location of the project, the project plan drawing, design depth, overdredge depth, disposal quantities and work details.
2. Previous dredging history including: type of work (i.e., maintenance or new work), date of last dredging operation, and quantity of sediment disposed.
3. Known or suspected dredge site contamination from either chemical and/or waste discharges and spills (industries, shipyards, oil terminals, major urban storm sewers, etc.) located on the map.

#### **C. Location of sampling areas**

1. Location of sampling areas within the project site.
2. Rationale for selection of the proposed sampling sites
3. Exact positions of each of the sediment sample sites and the reference site station (e.g., latitude and longitude, LORAN-C, state coordinate system, or Geographical Positioning System).
4. The model of the positioning equipment used in the sampling program, accuracy, precision of equipment, and a discussion of calibration procedures.

#### **D. Materials and Methods.**

1. Field sampling and sample handling procedures.
2. References for laboratory protocols for physical, chemical, bioassay, and bioaccumulation analyses including:
  - a. EPA method numbers and other EPA approved methods that do not have specific numbers.

- b. Method detection limits and references used for sediment and biological analyses.
- c. Test species used in each test, the supplier or collection site for each test species, the age of the organisms, and QA/QC procedures for test species.
- d. Location of reference and control sediment samples, QA/QC procedures and certification that the control sediment is free of contaminants.
- e. Source of seawater used in all biological tests and certification that the seawater meets the criteria for seawater defined in Chapters 11 and 12 of the 1991 Green Book.
- f. Bioassay testing procedures and QA/QC information.
- g. Statistical analysis procedures.

#### E. Final Results

- 1. Data summary tables (either typed or computer output)
- 2. Copies of the final raw data sheets that have been certified accurate

#### F. Discussion and Analysis of Data

- 1. Comparisons and contrasts with historical data from the proposed dredging site.
- 2. Statistical comparisons between the test sediments including procedures used and the rationale for their use.
- 3. An analysis and discussion of the suitability of the proposed dredged material for ocean disposal, by comparing tests results with those of a reference site, and by evaluating compliance with the EPA Ocean Dumping Regulations (40 CFR Part 227).

G. References. References used in the field sampling program, laboratory and statistical data analyses, as well as, historical data used in site comparisons.

H. Detailed Quality Assurance/Quality Control Plans and Information (EPA and CE 1991, Chapter, 14). Including the following:

- 1. Personnel Qualification.
- 2. Facilities layout, equipment and supplies.
- 3. Sample collection, handling and tracking.
- 4. Tests protocols and standard operating procedures for sediment and biological analyses

5. Documentation, record keeping, data validation and archiving.
6. Chemical quality control, biological quality control and reference toxicant testing.

## Appendix F

### **Report form for test conditions and test acceptability criteria for water-column acute toxicity, whole-sediment acute toxicity, and bioaccumulation.**

1. Test species:
2. Test type:
3. Culture temperature (° C):
4. Light quality (i.e., type):
5. Light intensity ( $\mu\text{E}/\text{m}^2/\text{sec}$ ):
6. Photoperiod (hr light and dark/day):
7. Test chamber size (250 ml minimum):
8. Test solution volume (200 ml minimum):
9. Renewal of test solutions:
10. Age of test organisms:
11. No. of organisms per test chamber:
12. Feeding regime:
13. Test chamber cleanings:
14. Test chamber aeration:
15. Dilution series:
16. Test concentrations:
17. Endpoint (e.g., lethality):
18. Sampling and sample holding requirements:

19. Sample volume required:

20. Test acceptability criterion:

21. Tissue weight, if this is a bioaccumulation test (wet wt., 30 grams minimum).



## **Appendix E**

### **Reference Site Locations in CESAD<sup>a</sup>.**

The following are Designated Reference Sites for ODMDS bioassay and bioaccumulation testing in CESAD:

<b>District:</b>	<b>Reference Site Location:</b>
Mobile	Marsh Island, Alabama

<sup>a</sup> Additional reference sites will be added as they become available.

## Appendix D

Summary of test conditions and test acceptability criteria (If test conditions do not exist Appendix F should be filled out and approved by the CE District).

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR <i>Crassostrea virginica</i> larvae, ACUTE TOXICITY WATER COLUMN TESTS.</b>	
1. Test type:	Static non - renewal
2. Test duration:	96 hour
3. Temperature:	25°C ± 1°C for <i>Crassostrea virginica</i>
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu\text{E}/\text{m}^2/\text{sec}$ (50-100 foot-candles) (ambient laboratory levels)
6. Photoperiod:	16 light/8 dark
7. Test chamber size:*	500 ml (minimum)
8. Test solution volume:*	500 ml (minimum)
9. Renewal of test solutions:	None
10. Age of test organisms:	Less than 4 hours
11. No. of organisms per test chamber:	7,500 - 15,000
12. No. of replicate chambers per concentration:	Minimum is 3
13. No. of organisms per concentration:	22,500 - 45,000
14. Feeding requirements:	Feeding not required
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
17. Dilution water:*	18-32 ppt ± 1ppt; natural seawater or suitable artificial seawater prepared with Milli-Q or equivalent deionized water

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR <i>Crassostrea virginica</i> larvae, ACUTE TOXICITY WATER COLUMN TESTS.</b>	
18. Test concentrations:	100% ambient water and a control
19. Dilution series:	100%, 50%, 10% of the dredged material elutriate
20. Endpoint	Shell development (significantly different from control)
21. Sampling and sample:	Grab samples are to be used holding requirements within 36 hours of collection
22. Sample volume required:	1 l/site
23. Test acceptability: *	≥70% or greater survival and 70% or greater shell development

\* Protocol dependent

**Reference:** ASTM 1989. E 724-89. Standard guide for conducting static acute toxicity tests starting with embryos of four species of saltwater bivalve molluscs. Annual Book of ASTM Standards Vol., 11.04. American Society for Testing and Materials, Philadelphia, PA.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR SHEEPSHEAD MINNOW, *Cyprinodon variegatus*, INLAND SILVERSIDE *Menidia beryllina*, ATLANTIC SILVERSIDE *Menidia menidia* AND TIDEWATER SILVERSIDE *Menidia peninsulae*, ACUTE TOXICITY WATER COLUMN TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	96 hour
3. Temperature:	20 or 25°C $\pm$ 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels)
6. Photoperiod:	16 light/8 dark
7. Test chamber size:	250 ml (minimum)
8. Test solution volume:	200 ml (minimum)
9. Renewal of test solutions:	None
10. Age of test organisms:	Sheepshead Minnow, 1-14 days $\pm$ 1 day; Silversides, 9-14 days $\pm$ 1 day
11. No. of organisms per test chamber:	Minimum is 10
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 50
14. Feeding requirements:	<i>Artemia</i> nauplii are made available while holding prior to test; add 0.2 ml <i>Artemia</i> nauplii concentrate at 48 hr intervals.
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	None unless DO concentrations fall below 40% saturation. Rate should not exceed 100 bubbles/minute

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  FOR SHEEPSHEAD MINNOW, <i>Cyprinodon variegatus</i>, INLAND SILVERSIDE  <i>Menidia beryllina</i>, ATLANTIC SILVERSIDE <i>Menidia menidia</i> AND TIDEWATER  SILVERSIDE <i>Menidia peninsulae</i>, ACUTE TOXICITY WATER COLUMN TESTS.</b>	
17. Dilution water:	Sheepshead minnow, 5-30 ppt $\pm$ 10%; Siversides, 5-32 ppt $\pm$ 10%; modified GP-2 Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	Three concentrations for site sediment; a reference concentration and a control
19. Dilution series:	100%, 50% and 10% for water column elutriate
20. Endpoint:	Lethality
21. Sampling and sample:	Composite samples are to be used within 14 days of the completion of the sampling period
22. Sample volume required:	Five gallons/project site and two gallons of sediment from reference site
23. Test acceptability:	$\geq$ 90% or greater survival in control treatment

**Reference:** EPA. 1991. Methods for measuring acute toxicity of effluents and receiving waters to freshwater and marine organisms. 4th edition. EPA/600/4-90/027.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR BROWN SHRIMP *Penaeus aztecus* AND WHITE SHRIMP *Penaeus setiferus*  
PINK SHRIMP *Penaeus duorarum*, ACUTE TOXICITY WHOLE SEDIMENT TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	96 hour
3. Temperature:	25°C ± 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	Ambient laboratory illumination
6. Photoperiod:	16 light/8 dark
7. Test chamber size:	80 l
8. Test solution volume:	60 l
9. Renewal of test solutions:	None
10. Age of test organisms:	None
11. No. of organisms per test chamber:	Minimum is 10
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 50
14. Feeding requirements:	None required
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
17. Dilution water:	30-35 ppt ± 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	NA
19. Dilution series:	NA
20. Endpoint:	Lethality

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR BROWN SHRIMP *Penaeus aztecus* AND WHITE SHRIMP *Penaeus setiferus*  
PINK SHRIMP *Penaeus duorarum*, ACUTE TOXICITY WHOLE SEDIMENT TESTS.**

21. Sampling and sample:	Composite samples are to be used within 14 days of the completion of the sampling period
22. Sample volume required:	Five gallons/project site and two gallons of sediment from reference site and two gallons control sediment
23. Test acceptability:	80% or greater survival in control treatment

**References:** Modified from the mysid water column test

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR OPOSSUM SHRIMP *Mysidopsis almyra*, *M. bahia*, and *M. bigelowi* ACUTE  
TOXICITY WATER COLUMN AND WHOLE SEDIMENT TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	96 hour
3. Temperature:	20°C $\pm$ 1°C or 25°C $\pm$ 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels).
6. Photoperiod:	16 light/8 dark
7. Test chamber size:	250 ml
8. Test solution volume:	200 ml
9. Renewal of test solutions:	None
10. Age of test organisms:	1-5 days $\pm$ 24 hours
11. No. of organisms per test chamber:	Minimum is 10
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 50
14. Feeding requirements:	<i>Artemia</i> nauplii are made available while holding prior to test; add 0.2 ml <i>Artemia</i> nauplii concentrate at 48 hr intervals.
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	None unless DO concentrations fall below 40% saturation. Rate should not exceed 100 bubbles/minute
17. Dilution water:	25-30 ppt $\pm$ 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	NA



**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR OPOSSUM SHRIMP *Mysidopsis almyra*, *M. bahia*, and *M. bigelowi* ACUTE  
TOXICITY WATER COLUMN AND WHOLE SEDIMENT TESTS.**

19. Dilution series:	100%, 50% and 10% for water column elutriate
20. Endpoint:	Lethality
21. Sampling and sample:	Composite samples are to be used within 14 days of the completion of the sampling period
22. Sample volume required:	Five gallons/project site and two gallons of sediment from reference site and two gallons control sediment for whole sediment toxicity
23. Test acceptability:	≥ 90% or greater survival in control treatment for water column toxicity or 80% or greater survival for whole sediment toxicity

**References:** EPA. 1991. Methods for measuring acute toxicity of effluents and receiving waters to freshwater and marine organisms. 4th edition. EPA/600/4-90/027.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR AMPHIPOD *Grandidierella japonica*, ACUTE TOXICITY WHOLE SEDIMENT  
TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	10 day
3. Temperature:	15-19°C $\pm$ 3°C
4. Salinity	30-35 ppt
5. Light quality:	Ambient laboratory illumination
6. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels).
7. Photoperiod:	Continuous light
8. Test chamber size:	1 l
9. Test solution volume:	2 cm sediment layer; seawater to the 950 ml level
10. Renewal of test solutions:	None
11. Age of test organisms:	Immature amphipods 3-6 mm length; no females carrying embryos
12. No. of organisms per test chamber:	20
13. No. of replicate chambers per concentration:	5
14. No. of organisms per concentration:	100
15. Feeding requirements:	Suspension of finely ground Tetramin and <i>Enteromorpha</i>
16. Test chamber cleaning:	Cleaning not required
17. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
18. Dilution water:	Clean (uncontaminated) seawater; reconstituted or natural seawater.
19. Test concentrations:	NA

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR AMPHIPOD <i>Grandidierella japonica</i>, ACUTE TOXICITY WHOLE SEDIMENT TESTS.</b>	
20. Dilution series:	NA
21. Endpoint:	Survival, emergence, reburial
22. Sampling and sample:	Sediment samples are to be used within 6 weeks of the completion of the sampling period
23. Sample volume required:	4 l/project site and two gallons of sediment from reference site and two gallons control sediment
24. Test acceptability:	≥ 80% or greater survival in control treatment

**References:** ASTM. 1991. Standard guide for conducting 10 day static sediment toxicity tests with marine and estuarine amphipods. pp. 1052-1075. Annual Book of ASTM Standards, vol, 11.04. American Society for Testing and Materials, Philadelphia, PA.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR AMPHIPOD *Rhepoxynius abronius*, ACUTE TOXICITY WHOLE SEDIMENT  
TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	10 day
3. Temperature:	15°C $\pm$ 3°C
4. Salinity:	30 to 35 ppt
5. Light quality:	Ambient laboratory illumination
6. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels).
7. Photoperiod:	Continuous light
8. Test chamber size:	1 l
9. Test solution volume:	2 cm sediment layer; seawater to the 950 ml level
10. Renewal of test solutions:	None
11. Age of test organisms:	Immature amphipods 3-5mm length; mixed sexes, no females carrying embryos
12. No. of organisms per test chamber:	20
13. No. of replicate chambers per concentration:	5
14. No. of organisms per concentration:	100
15. Feeding requirements:	Suspension of finely ground Tetramin and <i>Enteromorpha</i>
16. Test chamber cleaning:	Cleaning not required
17. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
18. Dilution water:	Clean (uncontaminated) seawater; reconstituted or natural seawater.
19. Test concentrations:	NA

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR AMPHIPOD <i>Rhepoxynius abronius</i>, ACUTE TOXICITY WHOLE SEDIMENT TESTS.</b>	
20. Dilution series:	NA
21. Endpoint:	Survival, emergence, reburial
22. Sampling and sample:	Sediment samples are to be used within 6 weeks of the completion of the sampling period
23. Sample volume required:	4 l/project site and two gallons of sediment from reference site and two gallons control sediment
24. Test acceptability:	≥ 80% or greater survival in control treatment

**References:** ASTM. 1991. Standard guide for conducting 10 day static sediment toxicity tests with marine and estuarine amphipods. pp. 1052-1075. Annual Book of ASTM Standards, vol, 11.04. American Society for Testing and Materials, Philadelphia, PA.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR AMPHIPOD *Eohaustorius estuarius*, ACUTE TOXICITY WHOLE SEDIMENT  
TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	10 day
3. Temperature:	15°C $\pm$ 3°C
4. Salinity:	2 to 28 ppt
5. Light quality:	Ambient laboratory illumination
6. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels).
7. Photoperiod:	Continuous light
8. Test chamber size:	1 l
9. Test solution volume:	2 cm sediment layer; seawater to the 950 ml level
10. Renewal of test solutions:	None
11. Age of test organisms:	Mature amphipods 3-5mm length; mixed sexes is acceptable
12. No. of organisms per test chamber:	20
13. No. of replicate chambers per concentration:	5
14. No. of organisms per concentration:	100
15. Feeding requirements:	None required
16. Test chamber cleaning:	Cleaning not required
17. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
18. Dilution water:	Clean (uncontaminated) seawater; reconstituted or natural seawater.
19. Test concentrations:	NA
20. Dilution series:	NA

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR AMPHIPOD <i>Eohaustorius estuarius</i>, ACUTE TOXICITY WHOLE SEDIMENT TESTS.</b>	
21. Endpoint:	Survival, emergence, reburial
22. Sampling and sample:	Sediment samples are to be used within 6 weeks of the completion of the sampling period
23. Sample volume required:	4 l/project site and two gallons of sediment from reference site and two gallons control sediment
24. Test acceptability:	≥ 80% or greater survival in control treatment

**References:** ASTM. 1991. Standard guide for conducting 10 day static sediment toxicity tests with marine and estuarine amphipods. pp. 1052-1075. Annual Book of ASTM Standards, vol, 11.04. American Society for Testing and Materials, Philadelphia, PA.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR AMPHIPOD *Ampelisca abdita*, ACUTE TOXICITY WHOLE SEDIMENT  
TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	10 day
3. Temperature:	20°C
4. Salinity:	28 to 35 ppt
5. Light quality:	Ambient laboratory illumination
6. Light intensity:	10-20 $\mu\text{E}/\text{m}^2/\text{sec}$ (50-100 foot-candles) (ambient laboratory levels).
7. Photoperiod:	Continuous light
8. Test chamber size:	1 l
9. Test solution volume:	4 cm sediment layer; seawater to the 950 ml level
10. Renewal of test solutions:	None
11. Age of test organisms:	Immature amphipods or mature females only
12. No. of organisms per test chamber:	20 to 30
13. No. of replicate chambers per concentration:	5
14. No. of organisms per concentration:	100 to 150
15. Feeding requirements:	Diatom culture in excess
16. Test chamber cleaning:	Cleaning not required
17. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
18. Dilution water:	Clean (uncontaminated) seawater; reconstituted or natural seawater.
19. Test concentrations:	NA
20. Dilution series:	NA



<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR AMPHIPOD <i>Ampelisca abdita</i>, ACUTE TOXICITY WHOLE SEDIMENT TESTS.</b>	
21. Endpoint:	Survival, emergence, reburial
22. Sampling and sample:	Sediment samples are to be used within 6 weeks of the completion of the sampling period
23. Sample volume required:	4 l/project site and two gallons of sediment from reference site and two gallons control sediment
24. Test acceptability:	≥ 80% or greater survival in control treatment

**References:** ASTM. 1991. Standard guide for conducting 10 day static sediment toxicity tests with marine and estuarine amphipods. pp. 1052-1075. Annual Book of ASTM Standards, vol, 11.04. American Society for Testing and Materials, Philadelphia, PA.

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR SAND WORM *Neanthes virens*, ACUTE TOXICITY SEDIMENT TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	10 day
3. Temperature:	20°C $\pm$ 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu$ E/m <sup>2</sup> /sec (50-100 foot-candles) (ambient laboratory levels).
6. Photoperiod:	12 light/12 dark
7. Test chamber size:	1 l minimum
8. Test solution volume:	200 ml of sediment and 200 ml of overlying water
9. Renewal of test solutions:	None
10. Age of test organisms:	2-3 weeks
11. No. of organisms per test chamber:	Maximum is 5
12. No. of replicate chambers per concentration:	3-5
13. No. of organisms per concentration:	15-25
14. Feeding requirements:	None required
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	Trickle flow
17. Dilution water:	20-35 ppt $\pm$ 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	Three concentrations for site sediment; a reference concentration and a control
19. Dilution series:	NA
20. Endpoint:	Survival

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR SAND WORM <i>Neanthes virens</i>, ACUTE TOXICITY SEDIMENT TESTS.</b>	
21. Sampling and sample:	Composite samples are to be used within 21 days of the completion of the sampling period
22. Sample volume required:	Two gallons of site, reference site and control sediment
23. Test acceptability:	≥ 80% or greater survival in control treatment

**References:** Modified from ASTM guidance document

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR SAND WORM *Neanthes virens*, SEDIMENT BIOACCUMULATION TESTS.**

1. Test type:	Static renewal
2. Test duration:	10 day (metals); $\geq 28$ day (nonpolar organics)
3. Temperature:	20°C $\pm$ 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu\text{E}/\text{m}^2/\text{sec}$ (50-100 foot-candles) (ambient laboratory levels).
6. Photoperiod:	12 light/12 dark
7. Test chamber size:	1 l minimum
8. Test solution volume:	200 ml of sediment and 200 ml of overlying water
9. Renewal of test solutions:	Weekly
10. Age of test organisms:	Adults of same year class
11. No. of organisms per test chamber:	Maximum is 5
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 25
14. Feeding requirements:	2 mg finely ground Tetramin or equivalent plus 0.5 mg finely ground alfalfa leaves twice weekly
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	Trickle flow (<100 bubbles/minute)
17. Dilution water:	20-35 ppt $\pm$ 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	NA
19. Dilution series:	NA

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR SAND WORM <i>Neanthes virens</i>, SEDIMENT BIOACCUMULATION TESTS.</b>	
20. Endpoint:	Survival, tissue residue
21. Sampling and sample:	Composite samples are to be used within 21 days of the completion of the sampling period
22. Sample volume required:	Two gallons of site, reference site and control sediment
23. Test acceptability:	≥ 90% or greater survival in control treatment

References: Modified from ASTM guidance document

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR BIVALVE *Macoma nastuta* , SEDIMENT BIOACCUMULATION TESTS.**

1. Test type:	Static renewal
2. Test duration:	10 day (metals); $\geq 28$ day (nonpolar organics)
3. Temperature:	12-16°C $\pm$ 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu\text{E}/\text{m}^2/\text{sec}$ (50-100 foot-candles) (ambient laboratory levels).
6. Photoperiod:	12 light/12 dark
7. Test chamber size:	1 l minimum
8. Test solution volume:	200 ml of sediment and 200 ml of overlying water
9. Renewal of test solutions:	Weekly
10. Age of test organisms:	Adults of same year class
11. No. of organisms per test chamber:	Maximum is 5
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 25
14. Feeding requirements:	2 mg finely ground Tetramin or equivalent plus 0.5 mg finely ground alfalfa leaves twice weekly
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	Trickle flow (<100 bubbles/minute)
17. Dilution water:	20-35 ppt $\pm$ 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	NA
19. Dilution series:	NA

<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR BIVALVE <i>Macoma nastuta</i> , SEDIMENT BIOACCUMULATION TESTS.</b>	
20. Endpoint:	Survival, Tissue residue
21. Sampling and sample:	Composite samples are to be used within 21 days of the completion of the sampling period
22. Sample volume required:	Two gallons of site, reference site and control sediment
23. Test acceptability:	≥ 90% or greater survival in control treatment

**References:** Modified from ASTM guidance document

**SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA  
FOR *Palaeomonetes spp.*, GRASS SHRIMP, ACUTE TOXICITY WATER COLUMN  
TESTS.**

1. Test type:	Static non - renewal
2. Test duration:	96 hour
3. Temperature:	25°C ± 1°C
4. Light quality:	Ambient laboratory illumination
5. Light intensity:	10-20 $\mu\text{E}/\text{m}^2/\text{sec}$ (50-100 foot-candles) (ambient laboratory levels).
6. Photoperiod:	16 light/8 dark
7. Test chamber size:	1 l
8. Test solution volume:	750 ml
9. Renewal of test solutions:	None
10. Age of test organisms:	None
11. No. of organisms per test chamber:	Minimum is 10
12. No. of replicate chambers per concentration:	Minimum is 5
13. No. of organisms per concentration:	Minimum is 50
14. Feeding requirements:	None required
15. Test chamber cleaning:	Cleaning not required
16. Test solution aeration:	None unless DO concentrations fall below 60% saturation. Rate should not exceed 100 bubbles/minute
17. Dilution water:	30-35 ppt ± 10%; modified GP-2, Forty Fathoms, or equivalent, artificial seawater prepared with Milli-Q or equivalent deionized seawater; or natural seawater.
18. Test concentrations:	NA
19. Dilution series:	100%, 50% and 10% for water column elutriate



<b>SUMMARY OF TEST CONDITIONS AND TEST ACCEPTABILITY CRITERIA FOR <i>Palaeomonetes spp.</i>, GRASS SHRIMP, ACUTE TOXICITY WATER COLUMN TESTS.</b>	
20. Endpoint:	Lethality
21. Sampling and sample:	Composite samples are to be used within 14 days of the completion of the sampling period
22. Sample volume required:	Five gallons/project site and two gallons of sediment from reference site and two gallons control sediment
23. Test acceptability:	≥ 90% or greater survival in control treatment

**References:** Modified from the mysid water column